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April 18, 2014

Mr. Dave Tomten
U.S. Environmental Protection Agency
1435 N. Orchard Street
Boise, ID 83706

RE: Ballard Shop Monitoring Well Sample Collection during P4's LTM Sampling - Final Rev 1 - 2014

Dear Dave:

This memorandum discusses the collection of additional groundwater samples from two wells located near the Ballard Shop during the Spring 2014 surface water (SW) and groundwater (GW) Long-Term Monitoring (LTM) Program. These two groundwater samples are being collected to address recommendations made in the *Remedial Investigation (RI) Report for P4's Ballard Mine – Draft Rev 0 [Ballard RI Report]* (MWH, 2014). It should be noted that these samples are a one-time collection event and, pending results, are not currently part of the LTM program. In an effort to maximize sampling efficiency, it is proposed that these samples will be collected during the Spring 2014 LTM sampling event.

The annual LTM Program is being performed at the P4 Production, L.L.C. (P4) inactive mine sites located north of Soda Springs, Idaho and in accordance with the requirements in the 2009 Administrative Settlement Agreement and Order on Consent/Consent Order for the Remedial Investigation/Feasibility Study (2009 AOC/ CO; RI/FS) with the US Environmental Protection Agency (USEPA) Region 10 and other named federal and state agencies. The P4 mine sites monitored include: Ballard, Henry, and Enoch Valley mines, collectively referred to as the Sites, which are the focus of the 2009 AOC/CO.

The two additional groundwater locations and samples are:

- **MBW011** - The base neutral (BN) fraction of semi-volatile organic compounds (SVOCs) analysis will be performed on groundwater collected from location MBW011 in the spring 2014 sampling round to address the AT comment #127 of the *Ballard RI Report*: “**Section 7.2.7, page 7-10, paragraph 1.** The detection of bis(2-ethylhexyl)phthalate in MBW011 appears to be anomalous as noted in the text but additional information should be added to the text to discount the detection as a lab or field contaminant. Expand the description or collect a new sample to justify dismissal of this detection.”
- **SB-07** - Tetrachloroethene (PCE) analysis will be performed on groundwater collected from temporary monitoring well SB-07 in the spring 2014 sampling round to address recommendations made in *Sections 7.2.7 and 7.4 of the Ballard RI Report* which state: “Groundwater collected in 2011 from SB-07 was slightly above the Idaho groundwater standard for PCE and it is recommended that this temporary monitoring well be resampled. Additional groundwater

characterization may be needed in the future if the presence of organic constituents is confirmed.”

It is anticipated that the Spring 2014 LTM sampling program for GW and SW will begin in May, based on snowpack and runoff conditions

The GW sample collection and analysis for these two locations will be performed according to the methods and procedures outlined within the *2011 Ballard Mine Shop Investigation Sampling and Analysis Plan-Revision 2 Final (2011 Ballard Shop SAP; MWH, 2011)*. **Figure 1** shows the two sample locations. The *2011 Ballard Shop SAP* is included as **Attachment 1** to this letter. Appendix A of the *2011 Ballard Shop SAP* contains a Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP).

The key elements of proposed sample collection are summarized on:

- **Table 1** - *2014 Ballard Shop Groundwater Analyte List*
- **Table 2** - *2014 Ballard Shop Groundwater Locations, Frequency, and Schedule*
- **Table 3** – *2014 Ballard Shop Spring Sample Tracker*

As shown on **Table 2**, no fall GW sampling is proposed for these two locations. Any additional sampling of these locations will be based upon results from this sampling event. P4 will submit all validated data and data validation summaries for the combine 2014 sampling effort within 120 days. In addition, the data will be included in the yearly Data Summary Reports submitted to the A/T per the 2009 AOC/CO SOW.

We appreciate your timely review of the proposed sampling program at the Ballard Shop area. If you have any questions or comments on this proposed sampling event, please do not hesitate to contact Rachel Roskelley at (208) 547-1248, or me at (801) 617-3250.

Best Regards,



Vance Drain, P.G.
Project Manager

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References

MWH, 2011 Ballard Mine Shop Investigation Sampling and Analysis Plan – Final Rev2

MWH, 2013 Remedial Investigation Report for P4's Ballard Mine – Draft Rev0

FIGURES



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TABLE 1
2014 BALLARD SHOP GROUNDWATER
ANALYTE LIST

Station ID	Fraction	Analytes (Analytical Method)
Groundwater		
SB-07	Unfiltered	PCE (EPA 8260B) Field Parameters ^a
MBW011	Unfiltered	BN fraction of SVOCs (EPA 8270C) Field Parameters ^a
Notes: ^a Field Parameters are listed on Table 3. BN – Base Neutral MBW – borehole monitor well PCE – tetrachloroethene SB – temporary monitor well SVOC – semi-volatile organic compound		

TABLE 2

2014 BALLARD SHOP GROUNDWATER LOCATIONS, FREQUENCY, AND SCHEDULE

			Location		Groundwater		
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Mine	Station ID	Station Description	Latitude	Longitude	Well Install Year	Groundwater System Monitored Screened Interval (ft. bgs)	Sample Schedule	Analyte List
Ballard Mine	SB-07 ^a	West of Ballard Shop near top of drive way into shop area.	42 49 32.93	111 29 34.47	2011	Alluvial 43-23	Spring	See Table 1
	MBW011 ^b	Ballard Creek	42 49 23.46	111 29 38.93	2008	Alluvial 15-10	Spring	See Table 1

Notes:

^aSB-07 is a temporary monitoring well near the Ballard Shop. See **Figure 1** for location. Well build specifications are: 2.0" sch 40 PVC casing, installed 43' below ground surface (bgs), 0.010" sch 40 PVC screen.

^aMBW011 is a borehole monitoring well (direct push well). Well build specifications are 1" sch 40 PVC casing, installed 15' bgs, pre-packed filter and sch 40 PVC screen.

TABLE 3
2014 BALLARD SHOP MONITORING WELL
SAMPLE TRACKER
 (Page 1 of 1)

					Lab Parameters - Analyte List ^b		Field Parameters										
Field Sample ID ^a	Location	Matrix	Filtered (check for yes)	QC Sample Type	PCE (EPA 8260B)	Semi-Volatile Organic Compounds Base Neutral (BN) Fraction only (EPA 8270C)	Specific Conductivity (µS/cm @ 25°C)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	Water Elevation (ft amsl)	Date Sampled	
GWMBW011-U	MBW011	Water	na	Primary		X	X	X		X	X	X	X	X	X		
GWSB-07-U	SB-07	Water	na	Primary	X		X	X		X	X	X	X	X	X		
B-GW-01-U	na	Water	na	B													

PCE - tetrachloroethene µS/cm microsiemens per centimeter
 % sat percent saturation °C
 mg/L Milligrams per liter ft³/sec
 mV Millivolts ftu

ID - identification

na - not applicable

QC - quality control

B – source water blank sample, to be taken once at the beginning of each sampling event and whenever new source water is used.

Primary QC sample indicates that it is the first samples collected.

^a Sample Identification will also include a date prefix reflecting the year and month the sample was taken. For example, a sample taken in May of 2014 would have a prefix of (1405) followed by the normal sample ID

^b The analyte list is presented in Table 1

MBW - Borehole Monitoring Well (direct-push pre-packed screen monitoring well)

ATTACHMENT 1
2011 BALLARD MINE SHOP INVESTIGATION SAMPLING AND ANALYSIS PLAN

BALLARD MINE SHOP INVESTIGATION

SAMPLING AND ANALYSIS PLAN

Revision 2
FINAL

MAY 2011

Prepared by:

MWH AMERICAS, INC.

Prepared for:

P4 PRODUCTION, LLC

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APPENDICES

Appendix A	Field Sampling Plan and Quality Assurance Project Plan
Appendix B	Health and Safety Plan Activity Hazard Analysis
Appendix C	Document Comments and Responses

ACRONYMS AND ABBREVIATIONS

A/T	Agencies and Tribes
bgs	Below Ground Surface
BLM	Bureau of Land Management (Department of Interior)
CO/AOC	Consent Order/ Administrative Order on Consent
COPC	Contaminant of Potential Concern
CSM	Conceptual Site Model
DOI	Department of the Interior
DQOs	Data Quality Objectives
FID	Flame Ionization Detector
e.g.	<i>exempli gratia</i> (Latin, for example)
FSP	Field Sampling Plan
ft	Feet
HASP	Health and Safety Plan
IDEQ	Idaho Department of Environmental Quality
i.e.	<i>id est</i> (Latin, that is to say; in other words)
MWH	MWH, Inc. (formerly Montgomery Watson Harza, Inc.)
P4	P4 Production, L.L.C.
PCB	Polychlorinated biphenyls
PID	Photoionization Detector
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation/Feasibility Study
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
TMP	Temporary Monitoring Point
TPH	Total Petroleum Hydrocarbons
Tribes	Shoshone-Bannock Tribes
USEPA	United States Environmental Protection Agency
USFS	United States Forest Service
UST	Underground Storage Tank
VOC	Volatile Organic Compound

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) has been prepared to describe the locations, rationale, and methods/procedures for the collection and analysis of soil and groundwater samples throughout the Ballard Mine shop area (the shop or shop area) located in the southwest portion of the Ballard Mine. This investigation is being performed as part of the characterization of the three historic P4 Production (P4) phosphate mines (i.e., the Ballard, Henry, and Enoch Valley Mines collectively known as “the Sites”) in southeastern Idaho. This SAP has been prepared in conjunction with, and will be attached to, the *Remedial Investigation/Feasibility Study Work Plan for P4’s Ballard, Henry, and Enoch Valley Mines* (the *RI/FS Work Plan*) for the comprehensive mine-specific RI/FS that is being conducted at the Sites.

This document has been prepared by MWH Americas, Inc. (MWH) on behalf of P4, in accordance with the requirements of the Administrative Settlement Agreement and Order on Consent/ Consent Order for Remedial Investigation/Feasibility Study (2009 CO/AOC; USEPA, 2009). The 2009 CO/AOC is a voluntary agreement between P4 and the United States Environmental Protection Agency (USEPA), the Idaho Department of Environmental Quality (IDEQ), the United States Department of Agriculture, Forest Service (USFS), the U.S. Department of the Interior (DOI), Bureau of Land Management (BLM), the Shoshone-Bannock Tribes (Tribes), collectively referred to as the “Agencies and Tribes” or A/Ts.

This SAP contains the key information for conducting the investigation of the shop area and will be provided to the field teams as they begin this work. The primary plans necessary for any SAP include a: Field Sampling Plan (FSP), Quality Assurance Project Plan (QAPP), and Health and Safety Plan (HASP). In this SAP, the FSP and QAPP components specific to the Ballard Mine Shop investigation are provided in Appendix A. A project-specific HASP hazard analysis for the Ballard Mine Shop Investigation is provided in Appendix B. In addition, the primary QAPP (and QAPP Addendum) and HASP for the overall RI/FS program are provided by reference in Appendices D6 and E of the *RI/FS Work Plan*. A key

component of the project planning process includes an evaluation of the Data Quality Objectives (DQOs) for the Ballard Mine Shop sampling effort. This analysis is presented in Section 2.0.

While the SAP components are often prepared as stand-alone documents, it needs to be recognized that this characterization is part of the overall characterization of the Sites within the RI/FS. Therefore, the more extensive *RI/FS Work Plan* components are not repeated herein (e.g., site background and data gaps analyses). The reader is referred to the following sections in the *RI/FS Work Plan* for a complete discussion of:

- Section 2.0 - Site background
- Section 3.0 - Existing data for each medium at each mine
- Section 4.0 - Data gaps identified for each medium at each mine

Section 4.0 includes information related to the need for the Ballard Mine Shop sample collection (especially in Section 4.4.1.1).

The objectives of the proposed Ballard Mine Shop sampling effort are presented in Section 1.1 below. Detailed information related to the location and rationale of individual sampling sites is presented in Section 2.0.

1.1 Background and Objectives

The current conceptual understanding and relevant data from the Ballard Site being investigated under the RI/FS are presented in Section 3.0 of the RI/FS Work Plan. All of the work completed to date has focused on inorganic (e.g., metals) analytes in media such as soil, sediment, surface water, and groundwater around the Sites. However, during the A/T review of the draft RI/FS Work Plan, the A/T identified as a data gap the Ballard Mine Shop area because it has not been investigated for potential organic contamination in soil or groundwater. As this shop was operated as a maintenance shop for heavy trucks and mining

equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines, there is a potential for spills and leaks (e.g., fuels or degreasers) resulting in potential organic contamination in shop area soils and groundwater.

During the active operation period, there may have been incidental spills/leaks of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining.

To assess the potential for contamination in the shop area, soil samples will be collected at locations around the perimeter of the shop and at other locations of potential contamination (e.g., former electrical facilities with transformers). However, not all potential source areas associated with the shop area can be readily investigated, and some sources may be unknown. Therefore, groundwater grab samples will be collected from the shallow alluvial aquifer in several boreholes to directly assess potential impacts to the alluvial groundwater system and to test for the presence of uninvestigated or unidentified sources.

The groundwater samples will be one-time grab samples collected from one upgradient and two downgradient locations in the shop area. In addition, one existing well MBW011 located further downgradient will be sampled. Piezometric data will be collected to help verify the groundwater flow direction.

1.2 Document Organization

The introduction including the background and project objectives are contained in Sections 1.0 and 1.1, above. Section 2.0 includes a detailed discussion of the project DQOs. Appendices necessary for the field teams to complete this specific scope of work include:

- Appendix A Field Sampling Plan (with necessary QAPP components)

- Appendix B Health and Safety Plan Activity Hazard Analysis
- Appendix C Document Comments and Responses

2.0 DATA QUALITY OBJECTIVES

The DQOs discussed in this section were used to guide the development of the components of this SAP (FSP and QAPP in Appendix A). The DQOs identify the quantity and quality of data that must be obtained to complete the Ballard Mine Shop investigation and to support the decision making process related to the RI/FS program.

2.1 DQO Presentation

The DQOs described herein are consistent with USEPA DQO guidance (USEPA, 2006a) and apply the following seven-step process:

1. State the problem
2. Identify the goals of the study
3. Identify information inputs
4. Define the boundaries of the study
5. Develop the analytic approach
6. Specify performance or acceptance criteria
7. Develop the plan for obtaining data

Within the DQOs, the principal study questions (from Step 2) have corresponding statements, as appropriate, in each of the remaining DQO steps. Outputs are given in each step and follow the 2006 DQO guidance. Refer to Section 1.1 for a general discussion of the Ballard Mine Shop sampling program objectives.

Each step of the DQO process defines criteria that will be used to establish the final data collection design. The first five steps are primarily focused on identifying qualitative criteria, such as:

- The nature of the problem that has initiated the study and a conceptual model of the environmental hazard to be investigated.

- The decisions or estimates that need to be made and the order of priority for resolving them.
- The type of data needed.
- An analytic approach or decision rule that defines the logic for how the data will be used to draw conclusions from the study findings (USEPA, 2006a).

The sixth step in the DQO process establishes acceptable quantitative criteria on the quality and quantity of the data to be collected, relative to the ultimate use of the data. For this characterization project, the data are primarily collected for the estimation of COPC concentrations in soils and groundwater around the Ballard Mine Shop.

In the seventh step of the DQO process, a data collection design is developed that will generate data meeting the quantitative and qualitative criteria specified at the end of Step 6. The output from this step is largely contained in the FSP/QAPP (provided as Appendix A). The measurement performance criteria for new data inputs are provided on Table 4-5 of Appendix A, and the comparison of analytical detection limits to the human health screening levels are provided on Tables 4-6 and 4-7 for soil and groundwater, respectively. Table 2-1 presents detailed information related to each of the seven DQO steps for the proposed Ballard Site shop area investigation discussed in this section of the document. Additional supporting information necessary to make informed decisions is provided in Section 2.2 below.

2.2 Supporting Information

Key factors that need to be considered in the DQO process are the site history and background information and the conceptual model, for helping formulate the problem statements (DQO Step 1.) Further information supporting the sample type, size and distribution is also presented in this section.

2.2.1 Site History and Background

The Ballard Mine shop was operated as a maintenance shop for heavy trucks and mining equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines. Since the Henry Mine closure in 1989, the shop has been used intermittently for storage. The former garage/shop building is still present. This shop is accessed through bay doors located on the east and west sides of the building (refer to Figure 2-1). This configuration was desirable for maintenance work because the haul trucks could be pulled in e.g., the west side of the building, and following maintenance pulled through the building exiting on the east side or back side of the building. The shop is built on a concrete foundation and has a concrete floor and its dimensions are approximately 120 feet by 120 feet. This building continues to be used by P4 and its mining contractor, Degerstrom Ventures, not as a maintenance shop, but now it is used to store vehicles, construction, maintenance materials, and other miscellaneous items. Off the southwestern corner of the shop building is an unused office building. Around the remainder of the shop area are several small sheds (both open and closed) that are used to store drill core, reclamation equipment, flammable materials and other miscellaneous items. The shop area also is used for a slag storage and two stockpiles are located in the area (refer to Figure 2-1). The slag is used for road repairs and also has been used as the road base in the shop area. Slag has been the base materials around the shop since the 1950's. However, based on appearance, a fresh layer has been laid down some time since the early 1990s.

Historical shop operations in the 1952 to 1989 time period included vehicle and equipment routine maintenance (e.g., oil and other fluid changes), overhauls, and welding. Organic materials that may have been associated with these activities conducted in the shop area include motor oil, grease, transmission fluids, hydraulic fluids, diesel fuel, gasoline, and degreasing solvents. The shop building contains both a grease pit and grated floor sump (refer Figure 2-1 for approximate locations within the building).

Transformers are present in two locations in the shop area. As shown on Figure 2-1, three transformers are located on an elevated platform just south of the shop building. In addition, to the west of the shop building, another larger transformer is located on a fenced,

concrete pad. There has been some limited sampling of the large transformers located on the pad to the west of the mine shop. As of 1995, the PCB levels were very low to not detectable in the transformer oils. However, there is no information on the three elevated transformers.

Three underground storage tanks (UST) were located near and off the northwest corner of the shop building (refer to Figure 2-1). Two of USTs stored 3,000 and 4,000 gallons of oil and the third tank stored 4,000 gallons of gasoline. These USTs were closed in October 1991 under the State of Idaho UST program (IDEQ, 1991). As part of the UST closure, total petroleum hydrocarbons (TPH) contamination was discovered. The TPH contamination was likely a result of surficial spills during refueling operations and underground pipe leakage. The contamination was found to extend out horizontally from the north side of the shop building in a pattern approximately 100 feet wide, 57 feet long and nine feet deep (Ankrum, 1991) as depicted on Figure 2-2. As approved by IDEQ, the contaminated soil was excavated in 1992 and land farmed until TPH levels were below IDEQ 100 mg/kg cleanup goal. The UST site was closed in 2003 according to the IDEQ website (<http://www.deq.idaho.gov/Applications/USTLUST/index.cfm?site=facility&facilitypk=3575>).

2.2.2 Conceptual Model

The primary components of the conceptual model for transport of organic constituents in soil and groundwater that support the DQOs are summarized as follows:

- Source – Past leaks and spills of petroleum fuels, degreasers, PCBs and other shop-related organic compounds could result in COPCs being present in subsurface soils immediately around the shop area and/or in shallow alluvial groundwater downgradient of the shop area. Current, on-going, leaks are not expected.
- Release mechanisms – The initial release mechanism was potentially leaks and spills from vehicles, tanks, floor drains, septic systems into site soils. Subsequent, and possibly currently ongoing, percolation of COPCs or leaching of vadose soils by infiltrating precipitation could result in impacts to the shallow alluvial groundwater.

- Exposure pathways – Potential exposure pathways include: (1) direct exposure to contaminated soil during excavation but, exposure to surface soils is not expected due to the presence of slag road base; and (2) consumption of contaminated groundwater (currently there is not a completed human health exposure pathway because domestic wells are not present in the area; however, the future well scenario needs to be considered).
- Receptors – Potential receptors include site workers during soil excavation during potential future construction or utility work, or humans or livestock from future exposure to groundwater extracted from domestic or agricultural wells, respectively. Currently there are no domestic wells in the shop area and is devoid of any forage for wildlife and of any flowing or standing water that would be attractive to ecological receptors or would provide habitat.
- Given the relatively remote rural location of the Ballard Shop Area, actual exposures are unlikely, but need to be evaluated. The site worker exposure scenario seems unlikely in that excavation of shop soils is not foreseen. If demolition of the buildings were to occur, the shop foundation may be broken up and removed. However, it also could be broken up and left in-place under revegetated cover soil. The most likely way the site worker scenario would be realized is if the shop area is utilized for vehicle maintenance again. If a new building is constructed in place of the current garage building, foundation construction would be likely and if so, soil excavation would occur. The potential groundwater exposure pathway includes an agricultural well (MAW0008) located in a position potentially downgradient of the shop area. In addition, a hypothetical future agricultural or domestic well also needs to be considered. Wildlife exposures to the shop area currently are unlikely (as described above), but will continue to be unlikely given the possible future uses of the Ballard Shop area.
- These conceptual model components will be re-evaluated, refined, and verified as the project advances to risk assessment. The primary objective of the study presented in this SAP is the characterization of the nature and extent of COPCs within the

Ballard Mine shop area. These data then will be available to facilitate the determination of potential risks, if any, to human health posed by shop COPCs.

2.2.3 Facility Maps

Soil and groundwater sampling locations for the Site are provided in support of the DQOs on Figure 2-2. The map includes the existing facilities and other features around the shop area.

2.2.4 Discussion of Sampling Locations and Rationale

The locations where the samples will be collected are presented here for the shop area. The planned locations are listed on Table 2-2 along with the associated rationale. The following discussion summarizes the rationale for the collection of soil and groundwater samples in the Ballard Shop area.

2.2.4.1 Soil Sampling Locations

Hydrocarbon and Solvent Investigation. Four soil sampling locations (SB-1 to SB-4) are proposed around the shop building outside of the concrete apron as shown on Figure 2-2 to investigate the potential for hydrocarbon and solvent contamination. Soil samples are proposed on the east and west sides of the building outside of the bay doors. The remaining samples will be placed on the north and south sides of the building. The soil sample locations may be adjusted based on visual reconnaissance of surficial staining on the concrete or other visual cues of contamination such as drain outfalls, etc. These locations may be moved due to problems with access (i.e., overhead electrical lines, gas lines, etc.).

PCB Investigation. At two soil boring locations (SB-5 and SB-6), soil samples will be collected at the location of current transformers to the south and west of the main shop building specifically for PCBs only (refer to Figure 2-2). One additional location is proposed to the west of the shop building near the transformer pad (SB-5) and the second location (SB-6) is adjacent to the three transforms located on an elevated platform just to the south

of the shop building. The soil sample locations may be adjusted based on visual evidence of surficial staining on the concrete or nearby soils.

2.2.4.2 Groundwater Sampling and Temporary Monitoring Locations

Groundwater grab samples will be collected from three temporary monitoring points (TMPs) installed around the main shop building (SB-1, SB-3, and SB-7). One groundwater grab sample will be collected from the soil boring installed on the north side of the building (SB-1). It is expected that this will be an upgradient location with respect to groundwater flow. However, this boring location is within the area investigated and remediated during the 1991 TPH project at the Ballard Shop. The second temporary TMP will be installed south of the shop itself (SB-3) and the third to the west (SB-7). It is expected that one or both of these locations are immediately downgradient of the shop area and would detect any contaminants in groundwater from the former USTs, as well as any leaks and/or spills inside the shop. Groundwater samples will be collected from these TMPs and analyzed to determine if there are chlorinated solvents or other risk-related organic chemicals (e.g., benzene, toluene, ethylbenzene, xylenes or polycyclic aromatic hydrocarbons) are present in the groundwater. These proposed locations are identified on Figure 2-2.

In addition, there is an existing alluvial groundwater monitoring well located downgradient of the shop area (MBW011) as depicted on Figure 2-2. This well also will be sampled for chlorinated solvents or other risk-related organic chemicals and used to evaluate groundwater potentially further downgradient from the shop area. Water levels from MBW011, the TMPs, as well as, MW-15A, MBW028, and MBW009 also will be measured and used to evaluate the shallow groundwater flow near the shop area.

2.2.5 Soil Sample Design Summary

Soil Investigation (Hydrocarbon and Chlorinated Solvent) - An investigation for hydrocarbon and chlorinated solvent-related organic compounds will be conducted throughout the Ballard Site shop area. The proposed soil boring locations where soil, and in some cases groundwater samples will be collected have been laid out around the shop

building to optimize the information gathered from each boring. Specific details regarding the methods and procedures to be used for the collection and analysis of the soil and groundwater samples are included in the FSP in Appendix A. This information is summarized below.

Four borings will be drilled using a hollow stem auger (or similar method) for the collection of soil and in some cases groundwater samples from locations SB-1, SB-2, SB-3, and SB-4. Over much of the shop area, the original ground surface is covered in slag that was emplaced since early in the shop's operation. Depending on the location, the slag could also be a coarse material that may allow much of a surface spill to infiltrate to the native soil. Therefore, the first sample will be collected at the native soil/slag interface, which is assumed to be approximately six to 12 inches below the ground surface (bgs).

A second sample will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil) or based on PID readings, odor, or staining of the soil contained in the split spoon sampler from that core interval. Following extraction of the soil core from the borehole and prior to collecting of the second soil sample, the soil in the split-spoon sampler will be screened with a Flame Ionization Detector (FID) or a Photo Ionization Detector (PID) and the result recorded. Should the FID/PID results indicate contamination in one section of the soil in the core sample, the second soil sample would be collected from that area and the depth bgs noted on the sample ID and in the log book. In addition, the soils in the top 5 feet of each boring will be continuously logged according to the Unified Soil Classification System (USCS). If a third sample is necessary as described below, then that borehole will be logged continuously to 10 feet bgs prior to soil sample collection.

If significant organic vapors are detected above background by the field instrumentation (i.e., PID or FID) at five feet bgs, then a third soil sample would be collected at a depth of nine to 10 feet bgs. Should significant contamination be detected in the 10 foot interval, then the borehole will be continuously cored until no PID readings, visual staining, or odors are observed or groundwater is encountered. A fourth and final soil sample will be collected

just beneath the identified contamination or just above the water table (i.e., capillary fringe) to confirm the vertical extent of contamination. More detail on the screening and collection of soil samples during the Ballard Mine Shop area investigation is provided in Appendix A, Section 3.2.2.

Therefore, a minimum of two samples and a maximum of four soil samples will be collected from each boring and submitted to the laboratory for analysis. As discussed further below, following collection of the soils samples, SB-1 and SB-3 will be extended to groundwater (estimated depth of 20-30 feet bgs) for collection of groundwater samples and water level data.

The soil samples will be packaged and submitted to the laboratory for volatile organic compounds (VOCs) (EPA Method 8260B) and semi-volatile organic compounds (SVOCs) (EPA Method 8270C) analyses. Samples for VOC analysis will be collected immediately upon retrieval of the sample interval, in accordance with U.S. EPA Method 5035A. Using an appropriate sample collection device (e.g., Encore sampler), approximately 5 grams (g) of sample will be collected and placed in a sample vial that contains preservative solution. Both VOCs and SVOCs samples will be placed in a cooler with ice and stored at 4°C for transport to a laboratory following chain-of-custody protocol. Again, specific details regarding the methods and procedures to be used for the collection and analysis of the soil samples are included in the FSP in Appendix A.

Groundwater Investigation (Hydrocarbon and Chlorinated Solvent) - Groundwater samples will be collected from three TMPs that will be installed in SB-1, SB-3, and SB-7, as well as, from alluvial well MBW011. As discussed above, soil samples will also be collected from two of these TMPs, SB-1 and SB-3. At SB-3, the soil will be continuously logged to its total depth to record the general stratigraphy of the shop area. All of the TMPs will be installed across the uppermost alluvial water table. Because it is assumed to be an unconfined groundwater system, the TMPs will be constructed using a 15-foot length of screen with approximately five feet above the water table and ten feet below the water table. The installation depth chosen for the screen is based on the assumption that the water table

might decrease substantially from spring's high to the low water level, which would be expected in the early fall.

The TMPs will be constructed similar to monitoring wells; however, their primary purpose will be for the measurement of water levels so that groundwater flow in the shop area can be evaluated. The TMPs will be abandoned as soon as possible after water level data has been collected (likely in the spring, summer and fall). However, the TMPs will be constructed so that if needed, they can be converted to monitoring wells.

Groundwater samples and water levels will be collected immediately following the installation of the TMPs. The TMPs will be surveyed and additional rounds of water levels will be collected in the summer and fall so that the groundwater flow direction(s) and any seasonal variation can be evaluated. This evaluation will be used to confirm the suitability of the groundwater samples collected at the TMP locations for evaluating possible groundwater contamination in shop area (i.e., the locations are in a downgradient position). If the groundwater is found to contain elevated levels of COPCs, the TMPs could be converted to permanent monitoring wells.

The three TMPs (SBs -1, -3, and -7) and MBW011 will be sampled each using new disposable polyethylene bailer. The groundwater samples will be analyzed for VOCs (EPA Method 8260B) and SVOCs (EPA Method 8270C). Specific details regarding the methods and procedures to be used for the construction of the TMPs, and collection and analysis of the groundwater samples at all locations at the Ballard Mine Site are included in the FSP in Appendix A.

Soil Investigation (PCBs) - Shallow soil samples also will be collected in the two identified transformer locations and analyzed for PCBs. Soil borings (SB-5 and SB-6) will be located next to the identified transformer areas to the west and south of the shop building as depicted on Figure 2-2. Soil samples will be collected at the native soil interface, which is assumed to be approximately six to 12 inches bgs. A second sample interval will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil).

Additional soil samples will be collected if contamination is observed in the second sample as described in Appendix A (FSP), Section 3.2.2. The soil samples will be analyzed for PCBs by EPA Method 8082.

2.2.6 Data Reporting

While this SAP is intended to help guide a specific investigation at the Ballard Mine Site, this investigation is supplemental to the overall P4 Site RI/FS. It is anticipated that the data collected as part of this investigation will be presented in the Ballard Mine RI Report and utilized in the risk assessment for the Ballard Mine Site. The raw data and data validation reports will be submitted to the A/T upon request when available. A data validation summary (DVS) consisting of validated data tables will be submitted to the A/Ts within approximately 90 days from the date of collection of the last sample from this field program.

3.0 REFERENCES

Ankrum, Keith. 1992. Site Assessment for Dravo Soda Springs Idaho.

IDEQ, 1991. Idaho Underground Storage Tank 30 Day Notice of Closure Form.

USEPA, 2006a. *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G-4. U.S. Environmental Protection Agency, Washington DC, EPA/240/B-06/001, February 2006.

TABLES

TABLE 2-1
BALLARD MINE SHOP INVESTIGATION DQOS

<p>Step 1 - State the Problem</p>	<p>All of the work completed to date at the Ballard Mine has focused on inorganic (e.g., metals) analytes in media such as soil, sediment, surface water, and groundwater around the Sites; however, during the A/T review of the draft RI/FS Work Plan, the A/T identified a data gap in that the Ballard Mine Shop area had not been investigated for potential organic contamination in soil or groundwater. As this facility was operated as a maintenance shop for heavy trucks and mining equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines there would be the potential for spills and leaks (e.g., fuels, degreasers, or PCBs) resulting in potential organic contamination.</p> <p>In addition, during the active operation period, there is documented contamination (i.e., 1991 UST Investigation) and may have been other incidental spills of PCBs, oil and other hydrocarbons or solvents (i.e., lubricants, fuel, degreasers etc.).</p> <p>Planning team, decision makers, and principal data users include P4 and the A/T.</p>
<p>Step 2 – Identify the Goals of the Study</p>	<p><u>Principal Study Question 1:</u></p> <p>Are shallow subsurface soils and alluvial groundwater impacted by potential leaks and spills of fuels, degreasers, or chlorinated solvents (VOCs and SVOCs) above a level of concern around the Ballard Mine Shop area?</p> <p><u>Alternative actions:</u></p> <ol style="list-style-type: none"> 1. No action. Soil and groundwater are not impacted by organic constituents above a level of concern. 2. Soil and groundwater data indicate impacts by organic contamination above a level of concern and therefore, additional sampling is necessary to delineate the nature and extent of contamination. 3. Soil and groundwater data indicate impacts by organic contamination, however, the contamination has been delineated and no additional sampling is warranted.

	<p><u>Decision/estimation statement:</u></p> <p>Decide whether sufficient data (spatial coverage) are available to adequately characterize the nature and extent of VOCs and SVOCs contamination in soil and groundwater around the Ballard Mine Shop.</p> <p><u>Principal Study Question 2:</u></p> <p>Are shallow subsurface soils impacted above a level of concern by potential leaks and spills of transformer oil containing PCBs below the transformer areas around the Ballard Mine Shop area?</p> <p><u>Alternative actions:</u></p> <ol style="list-style-type: none"> 1. No action. Soils are not impacted by PCBs above a level of concern. 2. Soil data indicate impacts by organic contamination above a level of concern and therefore, additional sampling is necessary to delineate the nature and extent of contamination. 3. Soil data indicate impacts by organic contamination, however, the contamination has been delineated and no additional sampling is warranted. <p><u>Decision/estimation statement:</u></p> <p>Decide whether sufficient data (spatial coverage) are available to adequately characterize the nature and extent of PCB contamination in soils around the Ballard Mine Shop.</p>
<p>Step 3 – Identify Information Inputs</p>	<p>The information inputs for the decision process includes the following items that may already exist or will need to be collected:</p> <ul style="list-style-type: none"> • Existing operational history and background information for the Ballard Mine Shop including the 1991 UST closure investigation reports, documentation, and associated agency correspondence. • List of soil and groundwater COPCs • Existing and refined conceptual site models. • Developed sample location maps (Figures 2-1 and 2-2). • Risk-based screening benchmarks for COPCs. • Soil and groundwater sample data to define the nature and extent and magnitude of potential releases.

<p>Step 4 – Define the Boundaries of the Study</p>	<p><u>Spatial boundaries:</u> Areas around the shop with the potential for organic contamination based on site history and/or visual reconnaissance.</p> <p><u>Vertical boundary:</u> Soil – Maximum depth of soil sampling will be approximately 10 feet bgs (i.e., the maximum depth of a possible foundation). However additional soil samples will be collected if the sample interval at 10 feet bgs indicates contamination (or at 5 feet bgs in the PCB borehole location).</p> <p>Groundwater – depth of shallow alluvial groundwater (approximately 20-30 feet bgs).</p> <p><u>Temporal boundary:</u> Soil and groundwater collection is planned for spring or summer 2011.</p> <p><u>Practical constraints:</u> Slag, rock, or other substrate conditions preventing soil sampling to depth.</p>
<p>Step 5 – Develop the Analytic Approach</p>	<p><u>Principal Study Question 1:</u> If shallow subsurface soils or alluvial groundwater data show impacts of VOCs and SVOCs above conservative risk-based benchmarks around the Ballard Mine Shop area and the proposed sampling points do not delineate the vertical or horizontal nature and extent of the impacts, then the need for additional characterization will be evaluated during a second phase of the investigation. If the soil or groundwater data do not show impacts above conservative risk-based benchmarks or the impacts are delineated, then no additional characterization is warranted.</p> <p><u>Principal Study Question 2 :</u> If shallow subsurface soils data show impacts of PCBs above conservative risk-based benchmarks around the transformer areas south and west Ballard Mine Shop and the proposed sampling points do not delineate the vertical or horizontal nature and extent of the impacts, then the need for additional characterization will be evaluated during a second phase of the investigation. If the soil data do not show impacts above conservative risk-based benchmarks or the impacts are delineated by the proposed investigation, then no additional</p>

	<p>characterization is warranted.</p>
<p>Step 6 – Specify Performance or Acceptance Criteria</p>	<p>The precision, accuracy, representativeness, comparability, and completeness criteria and the minimum detection limits will be used to evaluate the usability of analytical data in making decisions about the nature and extent of soil and groundwater contamination at locations around the Ballard Mine Shop area.</p> <p>All data must meet approved usability as defined in the RI/FS QAPP and QAPP Addendum in addition to data requirements specified in the Appendix A – the FSP.</p> <p>Specific details of the sampling design are set forth in the plan presented herein using the considerations that have been documented.</p>
<p>Step 7 – Develop the Plan for Obtaining Data</p>	<p>The sampling rationale and design based on existing historical knowledge of site operations are presented in Section 2.0 of this SAP. The sampling design will be further evaluated if new data indicates that the proposed locations and analytes are not sufficient to characterize the nature and extent of contamination. The measurement performance criteria for new data collection efforts are provided on Table 4-5 of the FSP/QAPP (provided as Appendix A of this SAP), and the comparison of analytical detection limits to the human health screening levels are provided on Tables 4-6 and 4-7 for soil and groundwater, respectively. The field and quality assurance requirements and methods/procedures are presented in the FSP/QAPP (Appendix A).</p>

TABLE 2-2
SOIL BORINGS AND MONITORING WELL
LOCATIONS AND RATIONALE

Station ID	Location	Sampling Summary	Rationale
Soil Boring Locations			
SB-1	North side of shop building	Soil and Groundwater for VOCs and SVOCs	Alluvial soil boring/ TMP upgradient of shop building, but located adjacent to former UST location and within the approximate limits of the 1991 TPH investigation. Will assist in determining if clean backfill was used to backfill the excavation (from the UST contamination) and if groundwater contamination exists beneath the former UST location. Measurements in this TMP will assist in evaluating the groundwater flow direction in the shop area.
SB-2	East side of shop building	Soil for VOCs and SVOCs	Outside of bay doors, centered along east side of the shop building. Will assist in determining if VOCs or SVOCs are in the soils where it is expected that the vehicles/machinery were exiting the building, and if any significant spills occurred in the building and spilled off the slab outside the bay doors.
SB-3	South side of shop building	Soil and Groundwater for VOCs and SVOCs	Alluvial soil boring/ TMP likely downgradient of shop building. Will assist in determining if there were any spills on the south side of the building and if VOCs or SVOC are found in groundwater downgradient of interior features (e.g., the floor drain and the oil pit). Measurements in this TMP will assist in evaluating the groundwater flow direction in the shop area.
SB-4	West side of shop building	Soil for VOCs and SVOCs	Outside of bay doors, centered along the shop building. Will assist in determining if VOCs or SVOCs are in the soils where it is expected that the vehicles/machinery were entering the building, and if any significant spills occurred in the building and spilled off the slab outside the bay doors.

TABLE 2-2
SOIL BORINGS AND MONITORING WELL
LOCATIONS AND RATIONALE

Station ID	Location	Sampling Summary	Rationale
SB-5	Transformer Area west of shop building	Soil for PCBs	Adjacent to transformers on a pad southwest of the shop building. Will assist in determining if PCBs were leaked into the shallow soils at this location. In most cases, if found, the PCBs remain in the shallow soils.
SB-6	Transformer Area south of shop building	Soil for PCBs	Adjacent (underlying if possible) to three transformers on elevated platform south of the shop building. Will assist in determining if PCBs were leaked into the shallow soils at this location. In most cases, if found, the PCBs remain in the shallow soils.
SB-7	West of shop building	Groundwater for VOCs and SVOCs	Alluvial soil boring/ TMP likely downgradient of shop building. Will assist in determining if there were any spills/ leaks of VOCs or SVOC at the Ballard shop which have been transported to groundwater and then dissolved and flowed downgradient from the source of contamination (e.g., the floor drain, the oil pit, and the USTs). Measurements in this TMP will assist in evaluating the groundwater flow direction in the shop area.
Monitoring Well Location			
MBW011	Southwest of shop building	Groundwater for VOCs and SVOCs	Existing alluvial monitoring well likely downgradient of the shop building. Will assist in determining if there were any spills/ leaks of VOCs or SVOC at the Ballard shop which have been transported to groundwater and then dissolved and flowed downgradient from possible sources of contamination (e.g., the floor drain, the oil pit, and the USTs). Measurements in this borehole well will assist in evaluating the groundwater flow direction in the shop area.

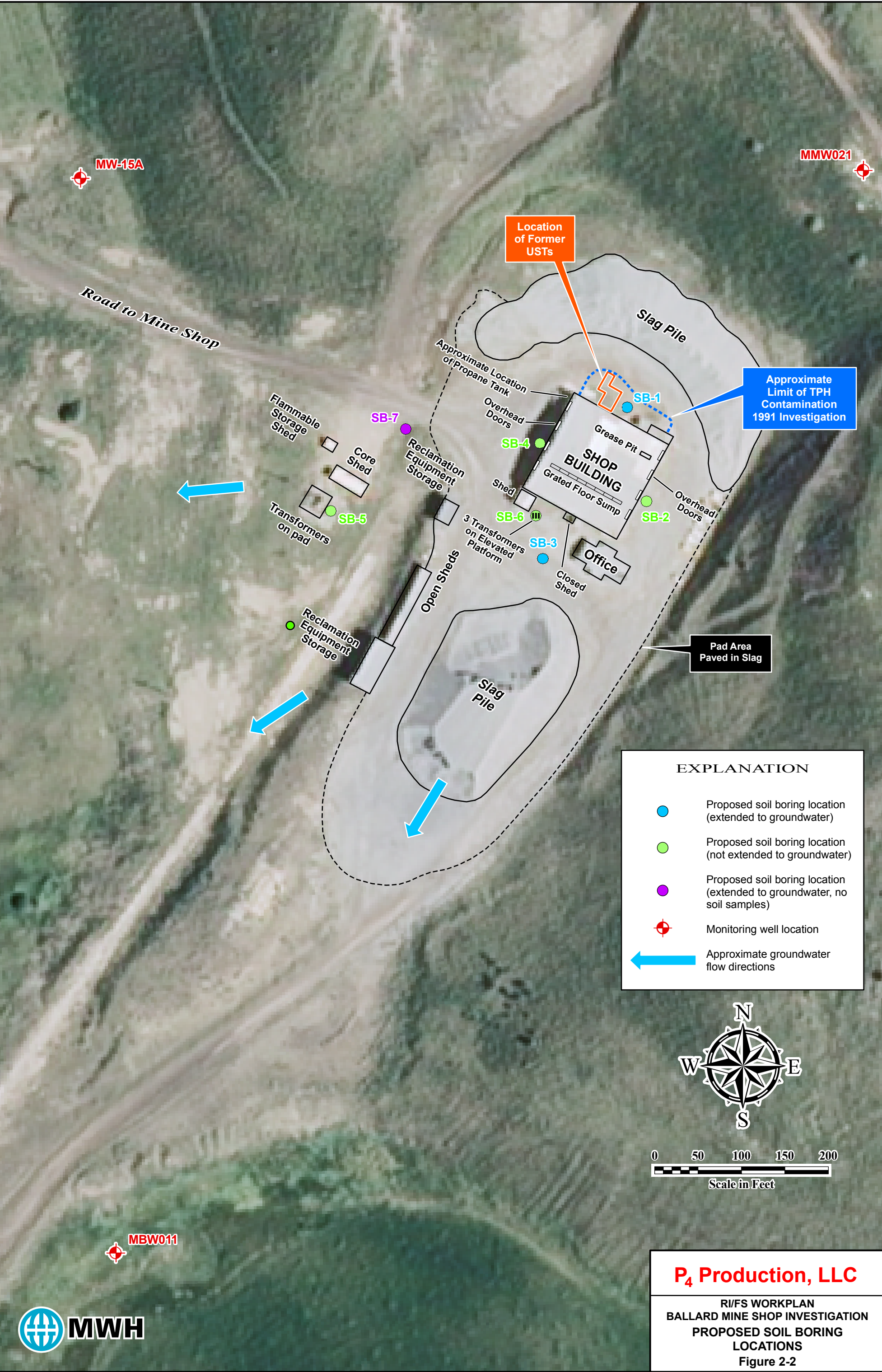
FIGURES



P₄ Production, LLC

PROJECT LOCATION	BALLARD, HENRY, AND ENOCH VALLEY MINES	
PROJECT	R/I/S WORK PLAN	
TITLE	BALLARD MINE SHOP LOCATION MAP	

	MWH	
	SHEET	FIGURE 2-1
	FILE NAME	Fig 2-1_Ballard Shop Site_winssetSPNAD27



APPENDICES

BALLARD SHOP SAP

APPENDIX A

FIELD SAMPLING PLAN

AND

QUALITY ASSURANCE PROJECT PLAN

APPENDIX A

BALLARD MINE SHOP INVESTIGATION

FIELD SAMPLING PLAN
AND
QUALITY ASSURANCE PROJECT PLAN

Revision 2
FINAL

MAY 2011

Prepared by:

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FSP/QAPP - APPROVAL PAGE

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APPENDICES

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ACRONYMS AND ABBREVIATIONS

A/T	Agencies and Tribes
AWRA	Area Wide Risk Assessment
cm	Centimeter
COPC	Contaminant of Potential Concern
DQI	Data Quality Indicator
DQOs	Data Quality Objectives
DSR	Data Summary Report
DVS	Data Validation Summary
dw	Dry Weight
EDD	Electronic Data Deliverable
e.g.	<i>exempli gratia</i> (Latin, for example)
FS	Feasibility Study
FSP	Field Sampling Plan
ft	Feet
ft ²	Square feet
GC/ECD	Gas Chromatography/Electron Capture Detector
GC/MS	Gas Chromatography/Mass Spectrometry
GPS	Global Positioning System
HHR	Human health risk
HASP	Health and Safety Plan
IDEQ	Idaho Department of Environmental Quality
i.e.	<i>id est</i> (Latin, that is to say; in other words)
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MDL	Method Detection Limit
mg/kg	Milligrams per Kilogram
MWH	MWH, Inc. (formerly Montgomery Watson Harza, Inc.)
P4	P4 Production, L.L.C.
PCB	polychlorinated biphenyl
QAPP	Quality Assurance Project Plan
RBS	Rapid Bioassessment Score
RPD	Relative Percent Difference

RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
TMP	Temporary Monitoring Point
TOC	Total Organic Carbon
USEPA	United States Environmental Protection Agency
USFS	United States Forest Service
VOC	Volatile Organic Compound

1.0 INTRODUCTION

This Field Sampling Plan and Quality Assurance Project Plan (FSP/QAPP) details the scope of work proposed for collection and analysis of soil and groundwater samples from the Ballard Mine Shop area. This FSP/QAPP is an attachment to the Ballard Mine Shop Investigation Sampling and Analysis Plan (SAP). The SAP presents the DQOs in Section 2.0 that have been developed to guide the sample collection program presented in this FSP/QAPP. Table 1-1 provides a cross reference to the 24 QAPP elements listed in the *Guidance for Quality Assurance Project Plans* (USEPA, 2002). Specific health and safety considerations are necessary for the work activities proposed during this upcoming investigation. An activity hazard analysis has been prepared for this investigation and is included in Appendix B of the SAP.

The Health and Safety Plan (HASP) is provided in Appendix E of the RI/FS Work Plan.

The FSP/QAPP is organized as follows:

- Section 1 – Introduction
- Section 2 – Program Background and Objectives provides a brief summary of background information related to the need for the Ballard Mine Shop area investigation, and objectives for the proposed sampling effort.
- Section 3 – Sampling Activities includes the processes used for selection of sample locations and rationale for their selection, equipment and procedures necessary to collect samples, and decontamination procedures that will be necessary during the field activities.
- Section 4 – Sample Handling includes discussion of sample designation, handling, shipping and sample analyses (including laboratory methods to be used), and quality assurance.
- Section 5 – Project Organization presents the project team, schedule, and deliverables
- Section 6 – References

2.0 PROGRAM BACKGROUND AND OBJECTIVES

This section provides brief background information related to the Ballard Mine Shop investigation. Additional program background and objective details are provided in Section 1.1 of the SAP and the *RI/FS Work Plan*. Because this facility was operated as a maintenance shop for heavy trucks and mining equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines there may have been incidental spills of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining.

To assess the potential for contamination in the shop area, soil samples will be collected at locations around the perimeter of the shop and at other locations of potential contamination (e.g., former electrical facilities with transformers). However, not all potential source areas associated with the shop area can be readily investigated, and some sources may be unknown. Therefore, groundwater grab samples will be collected from the shallow alluvial aquifer in several boreholes to directly assess potential impacts to the alluvial groundwater system and to test for the presence of contamination from uninvestigated or unidentified sources. Additional objectives for this sampling effort and rationale for individual sampling locations are provided in the SAP.

3.0 SAMPLING ACTIVITIES

Soil samples from borings will be collected to investigate the Ballard Shop area for hydrocarbons and other organic compounds. The surface around the shop is comprised of compacted slag which was used to cover the ground surface. Because of this, all soil sampling will be conducted using a hollow-stem auger (HSA) drill rig. All sampling will occur in the native materials below this slag road base. In addition, because shop operations date back to the 1950's and all potential source areas are not readily identified, grab groundwater samples will be collected in one upgradient and two downgradient groundwater locations in the shop area. In addition, one existing well MBW011 located further downgradient will be sampled. Piezometric data will be collected to help verify the groundwater flow direction.

3.1 Selection of Sampling Locations and Rationale

The specific locations where the samples will be collected are presented in Table 3-1 and the rationale for selection of each sample site is presented in Table 2-2 of the SAP. This information is summarized below.

3.1.1 Soil

Hydrocarbon and Solvent Investigation. Four soil boring locations (SB-1 to SB-4) are proposed around the shop building outside of the concrete apron as shown on Figure 3-1. Soil samples will be collected from these borings to investigate the potential for hydrocarbon and solvent contamination in the Ballard Shop area. The specific rationale for each proposed boring is discussed in Table 2-2 of the SAP. Soil borings are proposed on the west side and east sides of the building outside of the bay doors. The remaining two borings will be placed on the north and south sides of the former shop. The general intent of these borings is to assess the soils to approximately 10 feet below ground surface for the presence of spills and leaks that occurred around the shop or that flowed off the concrete shop floor and impacted the soil adjacent to the building. The soil boring locations may be adjusted

based on visual reconnaissance of surficial staining on the concrete surfaces or other visual cues of contamination such as drain outfalls, etc. These proposed boring locations also may be moved due to problems with access (i.e., overhead electrical lines, gas lines, etc.).

Polychlorinated Biphenyl (PCB) Investigation. At two soil boring locations (SB-5 and SB-6), soil samples will be collected adjacent to the location of current transformers to the south and west of the main shop building specifically for PCBs only (refer to Figure 3-1). One location is proposed to the west of the shop building near the transformer pad (SB-5), and the second location (SB-6) is adjacent to the three transforms located on an elevated platform just to the south of the shop building. It is assumed that any PCB-containing transformer oil was released through a surface spill or leak. The soil boring locations may be adjusted based on visual evidence of surficial staining on the concrete or nearby soils.

3.1.2 Groundwater

Groundwater grab samples will be collected from three temporary monitoring points (TMPs) installed in the soil borings that are proposed around the main shop building (SB-1, SB-3, and SB-7). One groundwater grab sample will be collected from the soil boring installed on the north side of the building (SB-1). It is expected that this will be an upgradient location with respect to groundwater flow. However, this boring location is within the area investigated and remediated during the 1991 TPH project at the Ballard Shop. The second soil boring will be installed south of the shop itself (SB-3) and the third to the west (SB-7). It is expected that one or both of these locations are immediately downgradient of the shop area and would detect any contaminants in groundwater from the former USTs, as well as any leaks and/or spills inside the shop. Groundwater samples will be collected from the TMPs installed in these borings and analyzed to determine if there are chlorinated solvents or other risk-related organic chemicals (e.g., benzene, toluene, ethylbenzene, xylenes or polycyclic aromatic hydrocarbons) are present in the groundwater. These proposed locations are identified on Figure 3-1.

The TMPs will be constructed similar to a monitoring well; however, their primary purpose will be the collection of water levels so that groundwater flow in the shop area can be evaluated. Groundwater samples and water levels will be collected immediately following the installation of the TMPs. The TMPs will be surveyed and additional rounds of water levels will be collected in the summer and fall so that the groundwater flow direction and any seasonal variation can be evaluated. This evaluation will be used to confirm the suitability of the groundwater samples collected at the TMP locations for evaluating groundwater flow in shop area (i.e., the locations are in a downgradient position). The TMPs will be abandoned within a year after water level data has been collected in the spring, summer and fall. However, if the groundwater is found to contain elevated levels of chlorinated solvents or other risk-related organic chemicals, as discussed earlier, the TMPs could be converted to permanent monitoring wells.

In addition, there is an existing alluvial groundwater monitoring well located downgradient of the shop area (MBW011) as depicted on Figure 3-1. This well also will be sampled for chlorinated solvents or other risk-related organic chemicals and used to evaluate groundwater potentially further downgradient from the shop area. Water levels from MBW011, as well as, MW-15A, MBW028, and MBW009 also will be measured and used to evaluate the shallow groundwater flow underlying the Ballard Mine Shop area.

3.2 Sample Collection Procedures

This section presents the site access, equipment, and procedures for the collection, handling, and analysis of each sampled medium. Samples will be analyzed according to the methods in Table 3-2. Where applicable, references to SOPs are provided.

3.2.1 Site Access, Logistics and, Safety

P4 has access to the Ballard Mine Shop building area. The A/T will be notified, at minimum, five business days prior to commencement of field activities. The MWH On-Site Safety Officer will notify the P4 Project Manager (Barry Koch) at minimum three days prior to working at a mine area. Such notification is necessary to arrange for any company-

specific safety training, and if necessary, to arrange for a company representative to accompany the crew to provide access to shop and equipment storage areas.

Any field equipment and samples stored will be stored at the Fox Hills Machine Shed, owned by P4. Equipment, supplies, and samples will be shipped and received from the Monsanto plant, in Soda Springs, in care of Barry Koch, P4. Additional sample handling and shipping information is presented in Section 4.2.

Safety procedures for the site investigation are described in the HASP located in Appendix E of the *RI/FS Work Plan* and in the Activity Hazard Analysis for this Program (Appendix B of the SAP). The mine-specific safety requirements involve a short training orientation for hazard recognition and avoidance. In the event that P4's corporate safety policy is stricter than the requirements of the HASP, those corporate safety requirements will take precedence.

3.2.2 Equipment and Procedures

Equipment and procedures for soil and groundwater sampling can vary considerably depending on conditions and equipment available. A HSA drill rig will be used to collect all soil and groundwater samples collected during the Ballard Shop investigation. Below, are descriptions of the procedures that will be followed when using this drill rig for soil, as well as, for groundwater sampling. The decision diagram in Figure 3-2 summarizes the sample collection rationale for both soil and groundwater samples.

Soil Investigation (Hydrocarbon and Chlorinated Solvent). The proposed soil boring locations have been laid out around the shop to optimize the soil and groundwater information gathered from each boring. Four borings will be drilled using a truck-mounted HSA rig (or similar method) for the collection of soil and in some cases groundwater samples from locations SB-1, SB-2, SB-3, and SB-4. Each borehole will be advanced within the alluvial material to the required depth. Soil samples will be collected using a Central Mine Equipment (CME) (or similar) split barrel sampling system or split-spoon samplers. In addition, the soils in the top 5 feet of each boring will be continuously logged according to

the Unified Soil Classification System (USCS). Soil samples from each split-spoon sampler will be:

- Visually inspected and logged (refer to SOP-1 for drilling and logging procedures) and,
- Tested for the presence of hydrocarbons/solvents using a photoionization or flame ionization detector (PID/FID)
- Used for soil sample collection at specific depth intervals.

Over much of the shop area, the original ground surface is covered in slag that was emplaced since early in the shop's operation. Depending on the location, the slag could also be a coarse material that may allow much of a surface spill to infiltrate to the native soil. Therefore, the first sample will be collected at the slag/native soil interface, which is assumed to be approximately six to 12 inches below the ground surface (bgs) and extend from this native ground surface to approximately one foot below the slag/soil interface.

Once the initial soil sample has been collected, upon retrieval from the borehole, the split spoon sampler will be laid on the vise and opened by the driller or geologist. The sample then will be cut open using a stainless steel knife to log the soil core and to collect PID/FID measurements by drawing the air in immediately above the sample face. If there is significant contamination, the PID will indicate it by elevated readings. The geologist will select the interval exhibiting the highest PID readings along the soil core for sample collection. However, if there are no significant readings, the field personnel will rely on staining or smell to define the appropriate sample interval. Should there be no significant PID readings, staining, or smell then the interval nearest the bottom of that sample will be selected for our soil sample.

A second sample will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil) or based on PID readings, odor, or staining of the soil contained in the split spoon sampler from that core interval. Following extraction of the soil core from the borehole and prior to collecting of the second soil sample, the soil in the split-spoon sampler will be screened with the PID/FID and the result recorded (as described above). Should the PID/FID results indicate more contamination in any one section of the soil in

the split-spoon or core barrel, the second soil sample preferentially would be collected from that area and the depth bgs noted on the sample ID and in the log book. If a third sample is necessary as described below, then that borehole will be logged continuously to 10 feet bgs prior to collection of the final soil sample. At SB-3, soils will be continuously sampled and logged from the ground surface to shallow groundwater table (total depth of approximately less than 20 to 30 feet bgs) so that the stratigraphy of the vadose zone can be better understood.

Based on a significant hit, as recorded above background by the field instrumentation (i.e., PID /FID) at the 5 foot bgs interval, a third soil sample would be collected at a depth of nine to 10 feet bgs and screened as described above before soil sample collection. Should significant contamination be detected in the 10 foot interval, then the borehole will be continuously cored until no PID readings, visual staining, or odors are observed or groundwater is encountered. A fourth and final soil sample will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.

Therefore, in each soil boring there will be a minimum of two soil samples and a maximum of four soil samples collected and submitted to the laboratory for analysis. As further discussed below, following collection of the soils samples, SB-1 and SB-3 will be extended to groundwater (estimated depth of 20 -30 feet bgs) for collection of groundwater samples and water level data.

The soil samples will be packaged and submitted to the laboratory for volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) analyses. Soil samples retained for VOCs analysis will be collected immediately upon retrieval from the core barrel or immediately after opening the split-spoon sampler, in accordance with U.S. EPA Method 5035A (refer to SOP-3). Using an appropriate sample collection device (e.g., Terracore sampler), approximately 5 grams (g) of sample will be collected from the designated interval (e.g., 4-5 feet below ground surface) and placed into sample vials that contains preservative solution as provided by the laboratory. Soil samples retained for SVOCs analysis will be

collected after the VOCs samples have been placed into sample vials and entered into the cooler.

The SVOCs samples will be grab samples collected from the appropriate samples interval using new or decontaminated stainless steel spoons or scoops and will be placed into new, appropriately sized sample jars provided by the laboratory. Both VOCs and SVOCs samples will be placed in a cooler with ice and stored at 4°C for transport to a laboratory following chain-of-custody protocol. Analytical parameters and methods to be analyzed are listed in Table 3-2. Specific details regarding the methods and procedures to be used for the collection and analysis of the soil samples are presented in SOP-1, SOP-2 and SOP-3 in Appendix A of this FSP/QAPP.

Groundwater Investigation (Hydrocarbon and Chlorinated Solvent). A HSA rig will be utilized to install TMPs in SB-1, SB-3, and SB-7. At SB-3, soils will be sampled continuously from the ground surface to groundwater (total depth of approximately 20 to 30 feet bgs). Soils will be logged in general accordance with USCS protocol (refer to SOP-1). At SB-1 borings, soil sample intervals will be logged as described above and soils will not be logged from 10 feet bgs to total depth. At SB-7, no soil samples will be collected or logged, however, this location will be used to collect a groundwater sample and to monitor the shallow water table downgradient of the shop area.

The borings will be advanced using 4.25-inch inner/8-inch outer diameter auger and TMPs will be constructed of 2-inch Schedule 40 polyvinyl chloride (PVC). All of the TMPs will be screened across the uppermost alluvial water table. Because it is assumed to be an unconfined groundwater system, the TMPs will be constructed using a 15-foot length of screen (approximately five feet above the water table and ten feet below the water table). The installation depth chosen for the screen is based on the assumption that the shallow water table might decrease substantially from spring's high to the low water level, which would be expected in the early fall.

The blank PVC casing will extend from the top of the well screen to about two feet above the ground surface. The annular space between the PVC and the augered hole will be filled

with the appropriately sized silica sand from the bottom of the borehole to two feet above the top of the well screen. The typical well construction in average formation materials includes filter pack on the order of #3 Monterey sand size and 0.020 inch slotted screen. For finer formations, 0.010 inch slotted screen may be used with appropriately graded sand (e.g., 20/40). Hydrated bentonite will be placed above the silica sand to prevent downward migration of surface water. The casing, sand pack, and bentonite seal will be installed through the augers as the auger string is slowly withdrawn from the borehole as described in detail in Appendix A of this FSP/QAPP, SOP-1. No lubricants, circulating fluid, drilling mud, or other additives will be used during this program.

Groundwater grab samples and water levels will be collected immediately following the installation of the TMPs. The TMPs, as well as MBW011, will be sampled using new disposable polyethylene bailers. Field parameters (pH, specific conductivity, DO, and temperature, etc.) will be monitored during sampling. Groundwater samples will be collected for laboratory analysis of VOCs and SVOCs.

The TMPs will be surveyed and additional rounds of water levels will be collected in the summer and fall so that the groundwater flow direction(s) and any seasonal variation can be evaluated. This evaluation will be used to confirm the suitability of the groundwater samples collected at the TMP locations for evaluating groundwater flow in shop area (i.e., the locations are in a downgradient position). If the groundwater is found to contain elevated levels of COPCs, the TMPs could be converted to permanent monitoring wells. However, if contamination is not found in the TMPs, they will be abandoned and backfilled in accordance with State regulations.

Specific analytical parameters and methods to be analyzed are listed in Table 3-2. Procedures for soil and groundwater sample handling and control, including chain-of-custody procedures and assurance and quality control (QA/QC), are presented in Section 4.0.

Soil Investigation (PCBs). Shallow soil samples also will be collected in the two identified transformer locations and analyzed for PCBs. Soil borings SB-5 and SB-6 will be located

next to the identified transformer areas to the west and south of the shop building as depicted on Figure 3-1. The HSA drill rig will be utilized to advance these two soil borings within the alluvial material to the required depth (refer to SOP-1). Soil samples will be collected with a CME (or similar) split barrel sampling system or split-spoon samplers. Samples will be collected at the native soil interface, which is assumed to be approximately six to 12 inches bgs. A second sample interval will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil).

Should visual contamination or odors be detected in the second sample interval, then the boreholes will be continuously cored until no contamination indicators are observed or groundwater is reached. A third and final soil sample will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.

The soil samples will be collected with a clean stainless steel spoon or scoop and placed in an appropriately-sized container as provided by the laboratory. Sampled soil intervals will be logged in general accordance with USCS protocol. The soil samples will be analyzed for PCBs according to the methods described on Table 3-2 and in Section 4.3.

3.2.3 Surveying

Each soil boring/TMP and other pertinent features observed in the field will be surveyed providing horizontal location within 0.1 feet. A water level measuring point will be established on the north side of the 2-inch PVC casing at each TMP. The vertical elevation of this point and that of the other soil boring locations will be surveyed to within 0.01 feet. All measurements will be referenced to the State Plane Coordinate System, North American Datum 1927.

3.2.4 Equipment Decontamination

Equipment used for collecting samples will be decontaminated prior to all sample acquisition activities. Sampling equipment (including split-spoon samplers, stainless-steel spoons, etc.)

will be cleaned and decontaminated prior to use and between each sampling location.

Equipment will be decontaminated as follows:

- Remove any excess rock fragments, soil, and vegetation from the sampling equipment
 - Wash the equipment in non-phosphate detergent (e.g., Crystal White, Alconox® or Liqui-Nox® solutions made as directed by the manufacturer)
 - Rinse with potable water
 - Rinse twice with deionized or distilled water
 - Allow equipment to air dry
 - Rinse water will be containerized pending receipt of soil and groundwater analytical data
- Hollow-stem augers and the associated drilling equipment (e.g., center plug) that contacts the soil will be decontaminated, as necessary, by pressure washing
 - Auger decontamination wash water will be containerized pending receipt of soil and groundwater analytical data

All decontamination water will be containerized and handled as IDW as discussed in the following section.

3.2.5 Investigation-Derived Waste (IDW)

Investigation generated IDW will include:

- Decontamination water from sampling equipment including hollow-stem augers
- Gloves, bailers, and other disposable equipment used to handle soil and groundwater
- Soil cuttings

Soil cuttings for boreholes not extending to groundwater (soil borings to 10 feet bgs) will be placed back in the borehole. Where there is excess soil (e.g., from soil borings where TMPs are installed), soil cutting may be containerized or placed on plastic and covered pending

completion of soil analyses. Soil cuttings from the various boreholes will not be comingled. Other IDW will be containerized in 55-gallon drums or other appropriate containers.

All IDW will be containerized or isolated from the environment pending the receipt of soil and groundwater analytical results. If the soil or groundwater is not contaminated, then the IDW may be handled as trash, or in the case of water or soil, disposed of on site. If contamination is identified, then the IDW will have to be handled appropriately depending upon the level and type of contamination present. The A/Ts will be appraised of IDW evaluation and final deposition.

3.2.6 Borehole Abandonment

Boreholes not extending to the water table (borings less than 15 feet bgs) will be backfilled with soil cuttings. For the boreholes extending to the water table, the Idaho Department of Water Resources (IDWR) abandonment regulations will be followed. IDWR regulation (IDAPA 37.03.09.12a) for well abandonment will be followed when TMPs are no longer necessary and are scheduled for abandonment. The general procedure for abandoning TMPs is as follows:

- If possible, the PVC casing will be pulled out of the borehole, otherwise it will be left in place.
- The borehole or casing will be sealed from the bottom up using bentonite pellets or chips, cement grout, or cement through the use of a tremie pipe.

3.3 Training Requirements

MWH field personnel will be trained in the requirements of the SAP in a project meeting prior to the initiation of field activity. All personnel will read the SAP documents prior to the start of field work, and will acknowledge completion of training at the time of the project meeting. Meeting notes and attendance sheets will be kept and forwarded to the project records. In addition, prior to conducting each day's sampling activities, the Field Team Leader, or designee will conduct a "tailgate" meeting with field staff to review field procedures and sampling requirements, in order to better ensure that samples are collected and handled according to FSP and QAP requirements. Tailgate meeting discussion subjects and attendees will be documented in the Field Logbook.

The Field Team Leader will maintain a hard copy of the current approved version of the entire SAP for ready-reference in the field vehicle or field office. Additionally, each field team will have a hard copy of the SAP.

3.4 Documentation and Records Requirements

3.4.1 Field Logbooks

Sample collection activities will be documented in permanently bound, page-numbered, weather-resistant field logbooks assigned to the Field Team Leader, or, if multiple sampling teams are used, to a designee in charge of each team. Each notebook will be identified to the project, task, and to the individual assigned custody of the logbook. For all sampling to be performed, the appropriate SOP, appended to the FSP, will also be employed. If logbook custody is transferred to another individual, such transfer will be noted in the logbook and signed and dated by both parties. All entries will be made in indelible ink; errors will be corrected by one single line through the text being revised, and all such corrections will be initialed and dated.

With the exception of the information contained in the appropriate SOP, governing the media to be sampled, bound field logbooks will be used to record the following information, as appropriate for the type of sampling being performed:

- Date, time, subjects, and attendees of daily tailgate training sessions
- Sample date, time, types, numbers, and quantities
- Sample container preservation steps performed
- Sample locations, including global positioning system (GPS) coordinates
- Numbers of associated photographs, with appropriate cross-references to the affected camera
- Sampling equipment used
- Decontamination steps performed
- Acknowledgements that chain-of-custody forms and express shipment information were properly completed

In addition, other ancillary information will be recorded, including:

- Time of arrivals/departures of MWH personnel and/or other visitors to the sampling site(s)
- Weather conditions
- Presence of livestock or wild game
- Time and subject of any incoming or outgoing telephone/radio contacts
- Any unusual events

The logbooks will be kept up to date on a daily basis; backup copies of each day's entries will be made on a weekly basis and forwarded separately to the project quality records, in addition to copies of all outgoing chains-of-custody and sample shipping documents.

3.4.2 Field Forms

In addition to the field logbooks, field forms will be required to be filled out by the sampling team conducting the sampling. All efforts will be made to fill out the information at the sampling location. Field forms for the sampling of soil and groundwater are used to

supplement the field logbooks. The appropriate forms are located in the applicable SOP (provided in Appendix A of this FSP/QAPP).

3.4.3 Chain-of-Custody Records

Documentation of sample custody must be maintained from the time the samples are collected through: receipt at the destination laboratory; sample homogenization, preparation, and analysis; data recording and reduction; data validation; and final release of laboratory analytical data. Initial information concerning sample collection will be recorded in the field logbook as described in Section 3.4.1. Information on the custody, transfer, handling, and shipping of samples will be recorded by field personnel on a project-specific chain-of-custody form for Microbac. A chain-of-custody form will be completed for each set of samples collected daily and will contain the following information:

- Sampler's signature and affiliation
- Project name and identification number
- Date and time of collection
- Sample identification number and matrix
- Analyses requested
- Number of containers
- Signature of persons relinquishing custody, dates, and times
- Signature of persons accepting custody, dates, and times
- Method of shipment
- Shipping papers/waybill identification number (e.g., Federal Express tracking number as identified on pre-printed packing labels)

A copy of each as-transmitted chain-of-custody form will be retained in the project records.

3.4.4 Analytical Laboratory Records

The contracted analytical laboratory will be responsible for preparing analytical laboratory reports that are reviewed and approved by the laboratory's QA manager prior to submittal to MWH.

Microbac's report will contain the following:

- A hard-copy data package with Stage 2B deliverables (see Section 3.4.4.1) and a scanned (e.g., “.pdf”) report with Stage 4 deliverables (see Section 3.4.4.2).
- Electronic data deliverable (see Section 3.4.4.3)

The hard-copy and scanned reports will be paginated and organized with a table of contents. The hard-copy deliverable will contain a cross reference that correlates the field identification as provided on the chain-of-custody document with the laboratory's sample identification. Results should be presented on a form equivalent to the United States Environmental Protection Agency's (USEPA or EPA) Contract Laboratory Program (CLP) “Form 1” (USEPA, 2005). Results from QC samples associated with each distinct analytical method are to be presented all together on QC summary sheets for ease of review. A Case Narrative will be provided for each analytical method. The Case Narrative will discuss any problem related to sample-receipt, corrective action taken by the laboratory, QC outliers or other problems, method deviations, and/or clarifications or anomalies observed by the laboratory.

Sample Results (CLP “Form 1” or equivalent) – This form contains all required data for field samples. The Form 1 will provide the following information:

- Field sample identification
- Laboratory sample identification
- Sample result(s) and appropriate units, method detection limit, and reporting limit. Concentrations equal to or greater than the method detection limit (MDL) must be reported. Concentrations between the MDL and reporting limit will be flagged as estimated (“J” flagged). Parameters that are not detected or present at a concentration less than the MDL are flagged as “U” and interpreted to be not detected at a value equal to or greater than the MDL. Do not report “not detected” (or “ND”).
- Sample collection and receipt dates
- Sample preparation date/time
- Analysis date/time
- Dilution factor

- Preparation batch number or identification
- Analysis batch number or identification
- Sample matrix and instrument
- For soil and vegetation sample, the samples will be reported as “dry-weight”

3.4.4.1 Summary or “Stage 2B” Data Deliverable Package for Organic Analysis

All summary forms need to be present, following the Form 1s, with clear association of the QC batch to each sample (on the CLP Form [USEPA, 2005] specified or equivalent and as applicable to the SW-846 method):

- Summary of all field sample results (as described above)
- Results of diluted and undiluted samples
- Sample results and preparation blank (Forms 1A, 1B, 1D, 1E and 1H)
- Surrogate compound recovery (Forms 2A, 2C, 2G, 2J, 2Q, and 2R)
- Matrix spike and matrix spike duplicate (MS/MSD) sample recovery and MS/MSD relative percent difference (RPD) (Forms 3A, 3B, 3C, 3D, 3J, and 3K)
- Laboratory control sample (LCS) recovery (Forms 3A, 3B, 3C, 3D as equivalent for LCS and Forms 3N and 3P)
- Method blank summary (Forms 4A, 4C, and 4F)
- Performance check for VOCs and SVOCs only (Forms 5A and 5B)
- Initial calibration data (Forms 6A, 6B, 6C, 6E, 6F, 6G, 6N, and 6P)
- Continuing calibration data (Forms 7A, 7B, 7C, 7E, 7F, 7G, and 7N)
- Internal standard area and retention time study for VOCs and SVOCs only (Forms 8A, 8C, 8D, and 8H) and analytical sequence for PCBs only (Form 8H)
- Identification summary for PCBs only (Form 10C)
- Sample log-in sheet (Form DC-1)
- Deliverables inventory sheet (Form DC-2)
- Case narrative
- Chain-of-custody

3.4.4.2 Full Raw Data or “Stage 4” Data Deliverable Package

The Full Raw Data Package includes all items specified for the Summary Data Package (Stage 2B), plus instrument raw data and/or documentation of the following:

- Results of diluted and undiluted samples
- Method blank
- Surrogate compounds
- Method blanks
- Performance check data
- Initial calibration data
- Continuing calibration data
- Laboratory duplicates
- LCS and matrix spikes (source, concentration, volume)
- Instrument identification
- Analysis date and time
- Full raw data print outs from instruments
- Full run log for each analysis
- VOCs and SVOCs to include: internal standard recoveries and tune data
- PCBs to include: identification data

3.4.4.3 Electronic Data Deliverable

Laboratory electronic data deliverables (EDDs) will contain detailed sample and laboratory QC sample data, including associations with QC batch sample results. Specifications for the EDDs are provided as Appendix B to this FSP/QAPP.

3.4.5 Documents and Records

Documents and records are defined as completed, legible documents which furnish objective evidence of the items or services, activities affecting quality or the completeness of data, and which are maintained for the specific project. These records will be organized and

managed in MWH's Bellevue, Washington program office and will include, at a minimum, the following:

- Original and backup copies of all bound field logbooks
- Field copies and original (laboratory) copies of all chain-of-custody documents
- Personnel training records (except that any medical monitoring program will be maintained in MWH's personnel files)
- Incoming and outgoing project correspondence (letters, telephone conversation records, faxes, and hard copies of e-mail messages)
- Copies of all laboratory agreements and amendments thereto
- Purchasing records for project supplies
- As-received laboratory data packages (hard copy and EDDs)
- Validated laboratory data packages
- All approved field change request (FCR) forms
- Draft and final versions of all reports and any associated presentation materials
- Draft and final delivered versions of the SI reports and its supporting procedures

3.4.6 Field Change Request Forms

Due to the conditions associated with field sampling activities, unexpected situations may occur that will require deviations or modifications to the requirements of the SAP. Other changes may be required by P4 during the course of this project. In such situations, the Program Manager may authorize the Field Team Leader or designee to undertake SAP modifications, provided that the scope of such modifications is discussed with the program Quality Manager and approved beforehand and documented on a FCR form. Each FCR will be uniquely numbered and will identify the project and task, the affected sections of the SAP or its supporting procedures, the scope of the requested variation, and the justification for its acceptance. At the Program Manager's discretion, the FCR may be forwarded to appropriate P4 representatives for review purposes prior to implementation. The field team leader will update field personnel of any changes.

4.0 SAMPLE HANDLING

This section presents the procedures for handling the samples once they have been collected and includes the labeling (designation), shipping, analysis and handling of the data generated from the analyses.

4.1 Sample Designation

Samples will be labeled with all necessary information on laboratory supplied labels using waterproof ink. Pre-printed labels will contain the following information:

- Site location
- Sample identification
- Method of preservation, if used
- Sample matrix

The date and time of sample collection and sampler's initials will be added to the label at time of collection.

Each sample will be assigned a unique identification number. This number will be coded according to sample location according to the following format for soil and groundwater samples:

AABB- XX-YYaa-b-c

where:

- **AA** indicates the year (two digits) the sampling event started
- **BB** indicates the month (two digits) the sampling event started
- **XX** denotes media type; media types are as follows:
 - SO: Soil
 - GW: Groundwater
- **YY** denotes the station type; station type which is SB for soil boring.

- **aa** denotes the specific station number/location.
- **b** denotes the depth interval in the case of the soil samples (e.g. 0.5 to 1' bgs) and in the case of groundwater denotes filtered (F) or unfiltered (U)
- **c** denotes the duplicate or replicate number (-blank shall indicate no replicate samples; if there are QA/QC replicate samples, then 1 and represent the blind duplicate or replicate samples).

As an example, sample number **1105-GW-SB01-U** describes a non-duplicated, unfiltered, groundwater sample collected at Soil Boring 1 in May of 2011. Sample **1106-SO-SB02-4to5'** indicates a standard soil sample collected from 4 to 5 feet below ground surface in Soil Boring 2 during June of 2011.

For equipment rinsate samples, the number will be identified as **AABB – ER – ZZ – bb**

- AA: Indicates the year (two digits) the sampling event started
- BB: Indicates the month (two digits) the sampling event started
- ER: Equipment Rinsate
- ZZ: Media type (soil, groundwater)
- bb: Rinsate number (01, 02, 03,... etc.)

4.2 Sample Handling and Shipping

Prior to sample collection, the field crew will ensure that adequate quantities of the following supplies and consumables are available in the field:

- Hand-held PID and calibration gas
- Sample containers (per Table 3-2), temperature blanks, and coolers
- Ice
- Rinsate water
- Personal protective equipment (e.g., gloves, suits, hard hats)
- Camera for photodocumentation
- Field notebooks
- Field forms (e.g., chain-of-custody, boring log, well development log)

Sample containers as provided by the laboratory will be placed on ice in an insulated cooler to $4 \pm 2^{\circ}\text{C}$. Insulated coolers will be provided by the contract laboratories or purchased locally. All samples will be stored in the coolers and handled as specified in the *P4 QAPP* and *QAPP Addendum*. All samples will remain in the coolers until the end of the day when all of the samples are shipped to the laboratory.

Samples will be shipped to the laboratories with blue ice or bagged standard ice in coolers with custody seals placed on the outside of the coolers (i.e., bridging the lid with the cooler side). Each cooler will be secured with packing tape and shipped via overnight Federal Express service to the appropriate laboratory. If possible, only one type of medium will be shipped in each cooler. MWH will fill out appropriate chain-of-custody forms supplied by the respective laboratory. The chain-of-custody will be included with the sample shipment, and copies of all chains-of-custody along with Federal Express waybills will be kept by MWH field personnel.

Samples will be sent to Microbac laboratory at the following address:

Microbac Laboratory
Ohio Valley Division
158 Starlite Drive
Marietta, OH 45750
(800) 373-4071 (phone)
(740) 373-4835 (fax)
Attn: Sample Receiving (Kathy Albertson)

Supplies including sample containers and coolers will be sent to the Monsanto Plant:

Monsanto Company
1853 HWY 34
Soda Springs, ID 83276
(208) 547-1439
Attention: Barry Koch

4.3 Sample Analysis

The target analyte lists for soil and groundwater samples are as follows:

- VOCs by EPA Method 8260B
- SVOCs by EPA Method 8270C
- PCBs by 8082

Tables 4-1 to 4-3 summarize the target analyte list and Microbac's method detection limits (MDLs) and reporting limits (RLs) for VOCs, SVOCs, and PCBs, respectively. Table 4-4 summarizes the samples to be collected and analytical laboratory and field methods to be performed.

Table 4-5 provides the performance measurement criteria for the data quality indicators (DQIs) for the project. The project human health risk (HHR) screening levels for soil and ground water are listed on Tables 4-6 and 4-7, respectively.

4.4 Field and Laboratory Quality Control Samples

The project-specific quality assurance plan for this sampling event is based on the procedures established in the Part 2 (the Quality Assurance Program Plan) of the *Comprehensive Site Investigation Sampling and Analysis Plan* (MWH, 2004) and the matrix-specific requirements provided in the *QAPP Addendum* (MWH, 2009a) for groundwater samples.

Field duplicates for each matrix will be collected at a rate of ten (10) percent of the number of primary samples, and matrix spike and matrix spike duplicate pairs will be collected at a rate of five (5) percent of the number of primary samples. One equipment rinsate will be collected each day if there is shared equipment between boreholes, and therefore a chance to cross-contaminate. In the instances where equipment rinsates need to be collected, one source water sample will be collected for the field event.

The relative percent difference (RPD) will be calculated for all values that are greater than their reporting limits. The data users will take into account the field replicate variability when assessing trends and/or decisions made with respect to field sample results. For soil samples, variability associated with the duplicate results will be a reflection of obvious

variability associated with the material being sampled, as well as any inherent variability in the sampling and analysis of the tested material. Therefore, the precision of duplicate samples will be used to document this variability, but will not be used to assess data usability with respect to comparisons of sample results to screening values. Variability for results in groundwater is not expected, so the RPD acceptance criterion is less than or equal to 20. Variability for results in soil is expected, so the RPD acceptance criterion is less than or equal to 35. Field sample results associated with RPDs greater than 20 for water field duplicate samples and 35 for soil field duplicate samples will be evaluated for impact on data usability.

Laboratory quality control samples and their requirements are detailed on Tables 4-8 through 4-10 for EPA Methods 8260B, 8270C, and 8082, respectively. The acceptance criteria for laboratory control sample (LCS) and laboratory control sample duplicate (LCSD) analysis are listed on Tables 4-11 through 4-13 for VOCs, SVOCs, and PCBs, respectively.

The laboratory's instrument maintenance schedule is summarized on Table 4-14.

4.5 Data Review, Verification, and Validation

The following definitions are provided in *Guidance for Quality Assurance Project Plans* (USEPA, 2002):

- Verification – the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual specifications.
- Validation – an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set.

Based on these definitions, the 3rd-party validator will be performing data verification of the sample, calibration, and QC data provided by the laboratory against the criteria specified in this project-specific QAPP. The validator will use the *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review* (USEPA, 2008) as a basis for performing data verification and qualification of data. Where appropriate, specific

references to the USEPA Functional Guidelines, as well as additional detail and/or deviation from that guidance, is detailed for EPA Methods 8260B, 8270C, and 8082 on Tables 4-10 through 4-12, respectively. The validator will document the data verification process on their in-house worksheets and summarize the results in data validation reports. Data validation reports will be consistent with the templates provided in Appendix C of this FSP/QAPP.

The data will be validated at the following two levels of effort per templates provided in appendix:

- Ten (10) percent of the data will be validated fully per EPA functional guidance (USEPA, 2008) to include raw data review. This level of review is referred to as USEPA Stage 2B verification/validation (USEPA, 2009)
- Ninety (90) percent of the data will be reviewed per data QC summaries only (no raw data reviews) to cover all QC parameters identified in the EPA functional guidance (e.g., initial calibration, initial calibration verification, continuing calibration, tuning, internal standard, as applicable to different methods). This level of review is referred to as USEPA Stage 4 verification/validation (USEPA, 2009).

The validator will use the following data qualifiers (“USEPA Flag”):

- U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample.
- J+ The result is an estimated quantity, but the result may be biased high.
- J- The result is an estimated quantity, but the result may be biased low.
- R The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample.
- UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

And the following “Reason Codes”:

- 1 Holding Time
- 2 Sample Preservation (including receipt temperature)
- 3 Sample Custody
- 4 Missing Deliverable

- 5 ICPMS or GC/MS Tune
- 6 Initial Calibration
- 7 Initial Calibration Verification
- 8 Continuing Calibration Verification
- 9 Low-Level Calibration Check Sample
- 10 Calibration Blank
- 11 Laboratory or Preparation Blank
- 12 ICPMS or ICP Interference Check Standard
- 13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
- 14 Laboratory Control Sample Precision
- 15 Laboratory Duplicate Precision
- 16 Matrix Spike or Matrix Spike Duplicate Recovery
- 17 Matrix Spike/Matrix Spike Duplicate Precision
- 18 ICPMS or ICP Serial Dilution
- 19 ICPMS or GC/MS Internal Standard
- 20 Field Replicate Precision
- 21 Equipment Rinsate Blank
- 22 Linear Range Exceeded
- 23 Other reason
- 24 Result is less than the MDC
- 25 Result is less than two times the error
- 26 Source Water Blank
- 27 Surrogate
- 28 Peak Resolution
- 29 Trip Blank

The validator will populate an MWH-supplied electronic data deliverable (EDD) with the following data:

- Field Header “USEPA Flag”: Populate with USEPA flags specified above and in template reports.
- Field Header “Reason Code”: Populate with all applicable Reason Codes as specified above and in template reports.
- Field Header “Final Result”: Populate with the final, qualified result, including any adjustment based on blank contamination.

The MWH Program Quality Manager will take the lead on validating the verified data. Data will be tabulated and assessed against the screening values. The reporting limits associated with non-detected values will be reviewed against the screening limits to evaluate whether the reported results are sufficiently sensitive as compared to the screening values. Results

that are estimated (J+ or J-) will be assessed for impact on data usability. Rejected results, as well as any sample that could not be collected or analyzed for any reason, will be evaluated, and a data-gap assessment will be performed and documented in the report.

4.6 Data Management

The individuals responsible for data management will include all personnel responsible for identifying, reporting, and documenting activities affecting data quality. The qualifications of individuals associated with data management activities will be commensurate with the level of expertise necessary to help ensure the intended level of evaluation.

All project files will provide a traceable record for all data management activities. The laboratory will maintain a project file that includes, but is not limited to, the following: formulas used, computer programs used, which data transfers are electronic or manual, validation steps. All data acquired electronically will be transferred and manipulated electronically to reduce errors inherent in manual data manipulation. Data entered, transferred, or calculated by hand will be spot checked for accuracy by someone who did not perform the original entries or calculations.

A project database will be designed to incorporate, at minimum, sample collection information (e.g., sample identification, location, date and time of sample collected, matrix) and laboratory analytical fields specified in the project EDD requirements (Appendix B of this FSP/QAPP). The EPA flags, Reason Codes, and final, qualified data will be uploaded from EDDs that the data validators will populate as discussed in Section 4.5.

4.7 Assessment and Response Actions

Assessment and response actions are typical field and laboratory performance audits. Neither a field audit nor laboratory audit is scheduled for the this field activity.

4.8 Reports to Management

The field team leader will summarize the daily sampling activities in a Daily Team Leader Progress Report form. This form requires the input of the following informations:

- Date activities occurred
- Identification of the field team leader and all other field sampling personnel
- Identification of subcontractors and vistors
- Summary of the work accomplished
- Identification of work planned or expected but not accomplished
- Description of activities planned for the next day of sampling

The daily progress report form is due to the P4 and MWH Project Managers at the end of day.

5.0 PROJECT ORGANIZATION

5.1 Project Team

Figure 1-1 of the *RI/FS Work Plan* presents the organization of the entire RI/FS project team. Contact information for each member of the project team is presented below in Table 5-1. The field team leader will submit a daily update to P4 and MWH project and task managers that contains a report of daily progress, any variances from planned work for the day, anticipated work for the next day, and any other problems or assistance required. A weekly update will be submitted to the A/T on-scene coordinator. All updates will be submitted via e-mail.

5.2 Project Schedule

- Ballard Mine Shop Investigation (1 event) – Between May 1 and June 30, 2011
- Data validation – within 60 days of receipt of laboratory data

5.3 Project Deliverables

While this SAP is intended to lead a specific investigation at the Ballard Mine Site, this investigation is supplemental to the overall P4 Site RI/FS. It is anticipated that the data collected as part of this investigation will be presented in the Ballard Mine RI Report and utilized in the risk assessment for the Ballard Mine Site. The raw data and data validation reports will be submitted to the A/T upon request when available. A data validation summary (DVS) consisting of validated data tables will be submitted to the A/Ts within approximately 90 days from the date of collection of the last sample from this field program.

Concentrations of groundwater and equipment rinsate blank samples will be expressed in terms of weight per unit volume (mg/L or µg/L). Concentrations of solid matrices (soil samples) will be expressed in terms of weight per unit weight of the dried sample from each sampling event (mg/kg or µg/kg dw). The number of significant figures in the field and

laboratory data presented in the final report will be consistent with the limits of uncertainty inherent in the measurement or analytical method. For the derivation of preliminary, risk-based benchmark concentrations, results are reported to one significant figure. Therefore, two significant figures will be retained for inputs to the risk model to minimize rounding error.

Table 4-5 notes that there are the method detection limits for several target compounds are greater than the human health screening levels for soil and groundwater. The specific compounds are identified with footnote “c” on Table 4-6 for soil (four SVOCs) and on Table 4-7 for groundwater (seven VOCs and 11 SVOCs). The uncertainty related to this will be addressed as part of the human health risk assessment.

6.0 REFERENCES

MWH. 2009a. *Final Quality Assurance Project Plan Addendum, Revision 2*. May.

United States Environmental Protection Agency (USEPA), 1991. *Management of Investigation-Derived Wastes During Site Inspections*. EPA/540/G-91/009. May 1991.

USEPA, 2002. *Guidance for Quality Assurance Project Plans*. EPA/240/R-02/009. Prepared by the USEPA Office of Environmental Information, Washington, DC. December.

USEPA, 2005. *USEPA Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration SOM01.1*. May

USEPA, 2008. *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*. EPA 540-R-08-01. Prepared by USEPA Office of Superfund Remediation and Technology Innovation, Washington, DC. June.

USEPA, 2009. *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use*. EPA 45-R-08-005. Office of Solid Waste and Emergency Response. January 13.

TABLES

TABLE 1-1

**CROSS REFERENCE FOR QAPP ELEMENTS
BALLARD MINE SHOP**

Item No.	QAPP Element	Document Location
Group A	Project Management	
A.1	Title and Approval Sheet	FSP/QAPP Approval Page
A.2	Table of Contents	SAP and FSP/QAPP TOCs
A.3	Distribution List	This SAP is an appendix the RI/FS Work Plan; the distribution list is presented in Section 1.3.3 of the Work Plan.
A.4	Project/Task Organization	SAP Section 1.0 and FSP/QAPP Section 5.1
A.5	Problem Definition/Background	SAP Section 1.1
A.6	Project/Task Description	SAP Section 2.2.2
A.7	Quality Objectives and Criteria for Measurement Data	SAP Section 2.1 and Table 2-1; FSP/QAPP Table 4-5
A.8	Special Training Needs/Certifications	FSP/QAPP Section 3.3
A.9	Documents and Records	FSP/QAPP Section 3.4
Group B	Data Generation and Acquisition	
B.1	Sampling Process Design	SAP Section 2.2.4
B.2	Sampling Methods	FSP/QAPP Section 3.2.2
B.3	Sample Handling and Custody	FSP/QAPP Sections 4.1 and 3.4.3
B.4	Analytical Methods	FSP/QAPP Section 4.3
B.5	Quality Control	FSP/QAPP Section 4.4
B.6	Instrument/Equipment Testing, Inspection, and Maintenance	FSP/QAPP Table 4-14
B.7	Instrument/Equipment Calibration and Frequency	FSP/QAPP Tables 4-8, 4-9, and 4-10
B.8	Inspection/Acceptance of Supplies and Consumables	FSP/QAPP Section 4.2
B.9	Non-direct Measurements	Screening values listed on FSP/QAPP Tables 4-6 and 4-7
B.10	Data Management	FSP/QAPP Section 4.6
Group C	Assessment and Oversight	
C.1	Assessment and Response Actions	FSP/QAPP Section 4.7
C.2	Reports to Management	FSP/QAPP Section 4.8
Group D	Data Validation and Usability	
D.1	Data Review, Verification, and Validation	FSP/QAPP Section 4.5
D.2	Verification and Validation Methods	FSP/QAPP Section 4.5
D.3	Reconciliation and User Requirements	FSP/QAPP Section 4.5 and 5.3

TABLE 3-1
BALLARD MINE SHOP
SOIL AND GROUNDWATER SAMPLING LOCATIONS

Station Number	Feature	Sampling Summary	Location	
			Latitude	Longitude
Soil Boring Locations				
SB-1	North side of shop building	Soil and Groundwater for VOCs and SVOCs	42.825924756	111.491145598
SB-2	East side of shop building	Soil for VOCs and SVOCs	42.825626329	111.491067694
SB-3	South side of shop building	Soil and Groundwater for VOCs and SVOCs	42.825448310	111.491512494
SB-4	West side of shop building	Soil for VOCs and SVOCs	42.825813988	111.491522537
SB-5	Transformer Area west of shop building	Soil for PCBs	42.825604474	111.492421580
SB-6	Transformer Area south of shop building	Soil for PCBs	42.825583926	111.491540746
SB-7	West of shop building	Groundwater for VOCs and SVOCs	42.825862819	111.492097278
Monitoring Well Location				
MWB011	Southwest of shop building	Groundwater for VOCs and SVOCs	42.823262979	111.493369673

TABLE 3-2

**REQUIREMENTS FOR SAMPLE CONTAINERS, VOLUMES, PRESERVATION, AND HOLDING TIMES
BALLARD MINE SHOP**

Parameter(s)	Analytical Method	Sample Container	Preservation	Holding Time ^a
Volatile organics (VOCs) in water	SW8260B	3 x 40 mL glass VOA vial with Teflon-lined septum cap	4 ± 2°C; HCl to pH < 2	14 days; 7 days for water if unpreserved by acid.
VOCs in soil	SW8260B	2-40ml VOA vials with stir bar, 5mls of Sodium Bisulfate solution and tare weight 1-40ml VOA vial with 5mls of Methanol and tare weight Terracore sampler and 1 dry weight container (via 5035)	4 ± 2°C	14 days
Semivolatile organic compounds (SVOCs) in water	SW8270C	2 X 1 liter; glass amber bottle with Teflon-lined cap	4 ± 2°C	7 days until extraction and 40 days after extraction
Polychlorinated biphenyls (PCBs) in water	SW8082	2 X 1 liter; glass amber bottle with Teflon-lined cap	4 ± 2°C	7 days until extraction and 40 days after extraction
SVOCs and PCBs in soil	SW8270C and SW8082	8-ounce glass jar with Teflon-lined lid	4 ± 2°C	14 days until extraction and 40 days after extraction

HCl - hydrochloric acid

mL - milliliters

°C - degrees Celsius

^a From date of sample collection

TABLE 4-1

**VOLATILE ORGANIC COMPOUNDS
PROJECT REPORTING LIMITS
BALLARD MINE SHOP
(Page 1 of 2)**

Parameter	Compound	Water (µg/L)		Soil (µg/kg)	
		MDL	RL	MDL	RL
Volatile Organic Compounds (VOCs)	1,1,1,2-Tetrachloroethane	0.25	5.0	0.5	5.0
	1,1,1-Trichloroethane	0.25	5.0	0.5	5.0
	1,1,2,2-Tetrachloroethane	0.20	5.0	0.5	5.0
	1,1,2-Trichloroethane	0.25	5.0	0.5	5.0
SW8260B	1,1-Dichloroethane	0.125	5.0	1.0	5.0
	1,1-Dichloroethene	0.50	5.0	0.5	5.0
	1,1-Dichloropropene	0.25	5.0	0.5	5.0
	1,2,3-Trichlorobenzene	0.15	5.0	0.5	5.0
	1,2,3-Trichloropropane	0.50	5.0	1.0	5.0
	1,2,4-Trichlorobenzene	0.20	5.0	0.5	5.0
	1,2,4-Trimethylbenzene	0.25	5.0	0.5	5.0
	1,2-Dichloroethane	0.25	5.0	0.5	5.0
	1,2-Dichlorobenzene	0.125	5.0	0.5	5.0
	1,2-Dibromo-3-chloropropane	1.0	5.0	2.0	5.0
	1,2-Dichloropropane	0.20	5.0	0.5	5.0
	1,2-Dibromoethane (EDB)	0.25	5.0	0.5	5.0
	1,3,5-Trimethylbenzene	0.25	5.0	0.5	5.0
	1,3-Dichlorobenzene	0.25	5.0	0.5	5.0
	1,3-Dichloropropane	0.20	5.0	0.5	5.0
	1,4-Dichlorobenzene	0.125	5.0	0.5	5.0
	2,2-Dichloropropane	0.25	5.0	0.5	5.0
	2-Chlorotoluene	0.125	5.0	0.5	5.0
	4-Chlorotoluene	0.25	5.0	0.5	5.0
	Acetone	2.5	10	5.0	10
	Benzene	0.125	5.0	0.5	5.0
	Bromobenzene	0.125	5.0	0.5	5.0
	Bromochloromethane	0.20	5.0	1.0	5.0
	Bromodichloromethane	0.25	5.0	0.5	5.0
	Bromoform	0.50	5.0	0.5	5.0
	Bromomethane	0.50	5.0	1.0	10
	Carbon tetrachloride	0.25	5.0	0.5	5.0
	Chlorobenzene	0.125	5.0	0.5	5.0
	Chloroethane	0.50	10	1.0	10
	Chloroform	0.125	5.0	0.5	5.0
	Chloromethane	0.50	10	2.0	10
	cis-1,2-Dichloroethene	0.25	5.0	0.5	5.0
	cis-1,3-Dichloropropene	0.25	5.0	0.5	5.0
	Dichlorodifluoromethane	0.25	10	1.0	10
	Dibromochloromethane	0.25	5.0	0.5	5.0
	Dibromomethane	0.25	5.0	0.5	5.0
	Ethylbenzene	0.25	5.0	0.5	5.0
	Hexachlorobutadiene	0.25	5.0	0.5	5.0
	Isopropylbenzene	0.25	5.0	0.5	5.0
	m,p-Xylene	0.50	5.0	0.5	5.0
	Methylene chloride	0.25	5.0	1.0	5.0
	Methyl t-butyl ether (MTBE)	0.5	1.0	0.5	5.0
	MEK (2-Butanone)	2.5	10	2.5	10
	MIBK (4-methyl-2-pentanone)	2.5	10	2.5	10
	n-Butylbenzene	0.25	5.0	0.5	5.0

TABLE 4-1
VOLATILE ORGANIC COMPOUNDS
PROJECT REPORTING LIMITS
BALLARD MINE SHOP
(Page 2 of 2)

Parameter	Compound	Water (µg/L)		Soil (µg/kg)	
		MDL	RL	MDL	RL
	n-Propylbenzene	0.125	5.0	0.5	5.0
	Naphthalene	0.20	10	0.5	10
	o-Xylene	0.25	5.0	0.5	5.0
	p-Isopropyltoluene	0.25	5.0	0.5	5.0
	sec-Butylbenzene	0.25	5.0	0.5	5.0
	Styrene	0.125	5.0	0.5	5.0
	Trichloroethene	0.25	5.0	0.5	5.0
	tert-Butylbenzene	0.25	5.0	0.5	5.0
	Tetrachloroethene	0.25	5.0	0.5	5.0
	Toluene	0.25	5.0	0.5	5.0
	trans-1,2-Dichloroethene	0.25	5.0	0.5	5.0
	trans-1,3-Dichloropropene	0.50	5.0	0.5	5.0
	Trichlorofluoromethane	0.25	10	1.0	10
	Vinyl chloride	0.25	10	1.0	10

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

µg/kg – micrograms per kilogram

µg/L – micrograms per liter

MDL – method detection limit

RL – reporting limit

TABLE 4-2

**SEMIVOLATILE ORGANIC COMPOUNDS
PROJECT REPORTING LIMITS
BALLARD MINE SHOP
(Page 1 of 2)**

Method ^a	Compound	Water, µg/L		Soil, µg/kg	
		MDL	RL	MDL	RL
Semivolatile organic compounds (SVOCs) SW8270C	1,2,4-Trichlorobenzene	2.5	5.0	82.5	165
	1,2-Dichlorobenzene	2.5	5.0	82.5	165
	1,3-Dichlorobenzene	2.5	5.0	82.5	165
	1,4-Dichlorobenzene	2.5	5.0	82.5	165
	2,4-Dinitrotoluene	2.5	5.0	82.5	165
	2,6-Dinitrotoluene	2.5	5.0	82.5	165
	2-Chloronaphthalene	2.5	5.0	82.5	165
	2-Methylnaphthalene	2.5	5.0	82.5	165
	2-Nitroaniline	12.5	25	330	825
	3-Nitroaniline	12.5	25	330	825
	3,3'-Dichlorobenzidine	2.5	5.0	165	330
	4-Bromophenyl phenyl ether	2.5	5.0	82.5	165
	4-Chloroaniline	2.5	5.0	82.5	165
	4-Chlorophenyl phenyl ether	2.5	5.0	82.5	165
	4-Nitroaniline	12.5	25	330	825
	Acenaphthylene	2.5	5.0	82.5	165
	Acenaphthene	2.5	5.0	82.5	165
	Anthracene	2.5	5.0	82.5	165
	Benz (a) anthracene	2.5	5.0	82.5	165
	Benzo (a) pyrene	2.5	5.0	82.5	165
	Benzo (k) fluoranthene	2.5	5.0	82.5	165
	Benzo (b) fluoranthene	2.5	5.0	82.5	165
	Benzo (g,h,i) perylene	2.5	5.0	82.5	165
	Benzyl alcohol	2.5	5.0	82.5	165
	Bis (2-chloroethoxy) methane	2.5	5.0	82.5	165
	Bis (2-chloroethyl) ether	2.5	5.0	82.5	165
	Bis (2-chloroisopropyl) ether	2.5	5.0	82.5	165
	Bis (2-ethylhexyl) phthalate	2.5	5.0	82.5	165
	Butyl benzylphthalate	2.5	5.0	82.5	165
	Chrysene	2.5	5.0	82.5	165
	Di-n-butylphthalate	2.5	5.0	82.5	165
	Di-n-octylphthalate	2.5	5.0	82.5	165
	Dibenz (a,h) anthracene	2.5	5.0	82.5	165
	Dibenzofuran	2.5	5.0	82.5	165
	Diethyl phthalate	2.5	5.0	82.5	165
	Dimethyl phthalate	2.5	5.0	82.5	165
	Fluoranthene	2.5	5.0	82.5	165
	Fluorene	2.5	5.0	82.5	165
	Hexachlorobenzene	2.5	5.0	82.5	165
	Hexachlorobutadiene	2.5	5.0	82.5	165
	Hexachloroethane	2.5	5.0	82.5	165
	Indeno (1,2,3-cd) pyrene	2.5	5.0	82.5	165
	Isophorone	2.5	5.0	82.5	165
	n-Nitrosodiphenylamine	2.5	5.0	82.5	165
	n-Nitrosodi-n-propylamine	2.5	5.0	82.5	165
	Naphthalene	2.5	5.0	82.5	165
	Nitrobenzene	2.5	5.0	82.5	165

TABLE 4-2

**SEMIVOLATILE ORGANIC COMPOUNDS
PROJECT REPORTING LIMITS
BALLARD MINE SHOP
(Page 2 of 2)**

Method ^a	Compound	Water, µg/L		Soil, µg/kg	
		MDL	RL	MDL	RL
	Phenanthrene	2.5	5.0	82.5	165
	Pyrene	2.5	5.0	82.5	165
	2,4,5-Trichlorophenol	2.5	5.0	82.5	165
	2,4,6-Trichlorophenol	2.5	5.0	82.5	165
	2,4-Dichlorophenol	2.5	5.0	82.5	165
	2,4-Dimethylphenol	2.5	5.0	82.5	165
	2,4-Dinitrophenol	12.5	25	330	825
	2-Chlorophenol	2.5	5.0	82.5	165
	2-Methylphenol	2.5	5.0	82.5	165
	2-Nitrophenol	2.5	5.0	82.5	165
	4,6-Dinitro-2-methylphenol	12.5	25	330	825
	4-Chloro-3-methylphenol	2.5	5.0	82.5	165
	4-Nitrophenol	12.5	25	330	825
	Benzoic acid	10	20	330	5,000
	Pentachlorophenol	12.5	25	330	825
	Phenol	2.5	5.0	82.5	165

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

µg/kg – micrograms per kilogram

µg/L – micrograms per liter

MDL – method detection limit

RL – reporting limit

TABLE 4-3

**POLYCHLORINDATED BIPHENYLS
PROJECT REPORTING LIMITS
BALLARD MINE SHOP
(Page 1 of 1)**

Parameter	Compound	Water (µg/L)		Soil (µg/kg)	
		MDL	RL	MDL	RL
Polychlorinated biphenyls (PCBs) SW8082A	PCB-1016	0.25	0.5	8.25	16.5
	PCB-1221	0.25	0.5	8.25	16.5
	PCB-1232	0.25	0.5	8.25	16.5
	PCB-1242	0.25	0.5	8.25	16.5
	PCB-1248	0.25	0.5	8.25	16.5
	PCB-1254	0.25	0.5	8.25	16.5
	PCB-1260	0.25	0.5	8.25	16.5

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

µg/kg – micrograms per kilogram

µg/L – micrograms per liter

MDL – method detection limit

RL – reporting limit

SAMPLES TO BE COLLECTED
BALLARD MINE SHOP
 (Page 1 of 3)

[illegible]

TABLE 4-4

SAMPLES TO BE COLLECTED
BALLARD MINE SHOP
(Page 2 of 3)

Field Sample Identification	Location	Mine	Matrix	Filtered/ Unfiltered	QC Sample Type	Analytical Laboratory Parameter (Method)			Field Parameter									
						VOCs (EPA 8260B)	SVOCs (EPA 8270C)	PCBs (EPA 8082)	Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)	
1105-SO-SB05 (4 to 5)	SB-05	Ballard	Soil	NA	Primary			X										
1105-SO-SB05 (depth TBD ^a)	SB-05	Ballard	Soil	NA	Primary			X										
1105-SO-SB06 (0.5 to 1)	SB-06	Ballard	Soil	NA	Primary			X										
1105-SO-SB06 (4 to 5)	SB-06	Ballard	Soil	NA	Primary			X										
1105-SO-SB06 (depth TBD ^a)	SB-06	Ballard	Soil	NA	Primary			X										
1105-GW-SB07-U	SB-07	Ballard	Water	Unfiltered	Primary	X	X			X	X	X	X	X	X	x	X	X
1105-GW-SB07-U-MS	SB-07	Ballard	Water	Unfiltered	MS	X	X			X	X	X	X	X	X	x	X	X
1105-GW-SB07-U-MSD	SB-07	Ballard	Water	Unfiltered	MSD	X	X			X	X	X	X	X	X	x	X	X
1105-GW-MBW011-U	MBW011	Ballard	Water	Unfiltered	Primary	X	X			X	X	X	X	X	X	x	X	X
1105-ER-SO-01-U	na	na	Water	Unfiltered	Equip Rinsate	X	X											
1105-ER-SO-02-U	na	na	Water	Unfiltered	Equip Rinsate	X	X											
1105-ER-SO-03-U	na	na	Water	Unfiltered	Equip Rinsate			X										
1105-ER-SO-04-U	na	na	Water	Unfiltered	Equip Rinsate			X										
1105-ER-GW-01-U	na	na	Water	Unfiltered	Equip Rinsate	X	X											
1105-ER-GW-02-U	na	na	Water	Unfiltered	Equip Rinsate	X	X											
1105-SW-01-U	na	na	Water	Unfiltered	Equip Rinsate	X	X	X										

^a If there is visual staining observed or obvious odors and/or if field instrumentation (e.g., PID,FID) measurements indicate potential contamination at the 4 to 5 feet below ground surface (bgs) interval, then the borehole will be continuously cored until there are (a) no visual staining observed, obvious odors, and field instrument detections, or (b) groundwater is encountered. A third soil sample will be collected at either the 9 to 10 feet bgs interval, just beneath the identified contamination, or just above the water table to confirm the vertical extent of contamination.

TABLE 4-4

SAMPLES TO BE COLLECTED
BALLARD MINE SHOP
 (Page 3 of 3)

						Analytical Laboratory											
						Parameter (Method)								Field Parameter			
Field Sample Identification	Location	Mine	Matrix	Filtered/ Unfiltered	QC Sample Type	VOCs (EPA 8260B)	SVOCs (EPA 8270C)	PCBs (EPA 8082)	Conductivity (uS/cm)	Specific Conductivity (uS/cm)	pH	Dissolved Oxygen (% sat)	Dissolved Oxygen (mg/L)	Oxidation/Reduction Potential (mV)	Turbidity (ftu)	Water Temperature (°C)	Air Temperature (°C)

^b If there is visual staining observed or obvious odors and/or if field instrumentation (e.g., PID,FID) measurements indicate potential contamination at the 9 to 10 feet bgs interval, then the borehole will be continuously cored until there are (a) no visual staining observed, obvious odors, and field instrument detections, or (b) groundwater is encountered. A fourth soil sample will be collected at either just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.

B – source water blank sample, to be taken once per field effort

ER - equipment rinsate blank sample, to be taken once per field team per day, total ERs taken may not add up to what is accounted for here

MS - matrix spike

MSD - matrix spike duplicate

NA - not applicable

QC - quality control

SW- source water

TBD - to be determined

TABLE 4-5

**PROJECT PERFORMANCE MEASUREMENT CRITERIA
BALLARD MINE SHOP**

DQI	Criteria	Project-Specific Goal
Detection levels	The analytical detection levels should be less than the applicable screening criteria.	The achievable laboratory reporting limits and method detection limits for soil and groundwater samples are listed on Tables 4-1 through 4-3. Laboratory method detection limits are less than or equal to all screening levels except those footnoted with letter "c" on Tables 4-6 and 4-7.
Accuracy	Spiked target analytes should be recovered within the limits established for each target analyte.	Recoveries of target analytes spiked into laboratory control samples and matrix spike samples should be within the control limits specified on Tables 4-11 through 4-13 for those quality control samples.
Precision	Measured values of target analytes should be reproducible within the limits established for each target analytes.	<p>Relative percent differences (RPDs) measured between laboratory control samples and laboratory control sample duplicates & matrix spike and matrix spike duplicates should be within the control limits specified on Tables 4-8 through 4-10 for those quality control samples.</p> <p>The RPDs for field duplicate samples are less than or equal to 20 for water and less than or equal to 35 for soils.</p>
Completeness	Each sample that is planned to be collected should be collected, analyzed, reported, and validated for each target analyte as specified in Section 4.4, except where actual site conditions prevent collection of sample as planned.	A minimum of 90% of planned soil samples and 90% of planned groundwater samples will be collected. All target analytes will be tested, reported, and validated for each collected sample.

TABLE 4-6

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR SOIL
BALLARD MINE SHOP
(Page 1 of 4)**

Compound	Soil (mg/kg)		Screening Level (mg/kg)	Source ^b
	MDL	RL		
<i>Volatile Organic Compounds (VOCs) by 8260B^a</i>				
1,1,1,2-Tetrachloroethane	0.0005	0.005	1.9	B
1,1,1-Trichloroethane	0.0005	0.005	8,700	B
1,1,2,2-Tetrachloroethane	0.0005	0.005	0.56	B
1,1,2-Trichloroethane	0.0005	0.005	1.1	B
1,1-Dichloroethane	0.001	0.010	3.3	B
1,1-Dichloroethene	0.0005	0.005	240	B
1,1-Dichloropropene	0.0005	0.005	NA	NA
1,2,3-Trichlorobenzene	0.0005	0.005	49	B
1,2,3-Trichloropropane	0.001	0.010	0.005	B
1,2,4-Trichlorobenzene	0.0005	0.005	22	B
1,2,4-Trimethylbenzene	0.0005	0.005	62	B
1,2-Dichloroethane	0.0005	0.005	3.4	A
1,2-Dichlorobenzene	0.0005	0.005	1,900	B
1,2-Dibromo-3-chloropropane	0.001	0.010	0.0054	B
1,2-Dichloropropane	0.0005	0.005	0.89	B
1,2-Dibromoethane (EDB)	0.0005	0.005	0.0034	B
1,3,5-Trimethylbenzene	0.0005	0.005	780	B
1,3-Dichlorobenzene	0.0005	0.005	NA	NA
1,3-Dichloropropane	0.0005	0.005	1,600	B
1,4-Dichlorobenzene	0.0005	0.005	2.4	V
2,2-Dichloropropane	0.0005	0.005	NA	NA
2-Chlorotoluene	0.0005	0.005	1,600	B
4-Chlorotoluene	0.0005	0.005	5,500	B
Acetone	0.005	0.010	61,000	B
Benzene	0.0005	0.005	8.5	A
Bromobenzene	0.0005	0.005	300	B
Bromochloromethane	0.001	0.005	NA	NA
Bromodichloromethane	0.0005	0.005	0.27	B
Bromoform	0.0005	0.005	61	B
Bromomethane	0.001	0.010	7.3	B
Carbon tetrachloride	0.0005	0.005	0.61	B
Chlorobenzene	0.0005	0.005	290	B
Chloroethane	0.001	0.010	15,000	B
Chloroform	0.0005	0.005	0.29	B
Chloromethane	0.002	0.010	120	B
cis-1,2-Dichloroethene	0.0005	0.005	160	B
cis-1,3-Dichloropropene	0.0005	0.005	1.7	B
Dichlorodifluoromethane	0.001	0.010	180	B
Dibromochloromethane	0.0005	0.005	0.68	B
Dibromomethane	0.0005	0.005	25	B
Ethylbenzene	0.0005	0.005	36	A
Hexachlorobutadiene	0.0005	0.005	6.2	B
Isopropylbenzene	0.0005	0.005	NA	NA
m,p-Xylene	0.0005	0.005	3,480	A
Methylene chloride	0.001	0.005	11	B

TABLE 4-6

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR SOIL
BALLARD MINE SHOP
(Page 2 of 4)**

Compound	Soil (mg/kg)		Screening Level (mg/kg)	Source ^b
	MDL	RL		
Methyl t-butyl ether (MTBE)	0.0005	0.005	345	A
MEK (2-Butanone)	0.0025	0.010	28,000	B
MIBK (4-methyl-2-pentanone)	0.0025	0.010	5,300	B
n-Butylbenzene	0.0005	0.005	NA	NA
n-Propylbenzene	0.0005	0.005	3,400	B
Naphthalene	0.0005	0.010	50	A
o-Xylene	0.0005	0.005	3,480	A
p-Isopropyltoluene	0.0005	0.005	NA	NA
sec-Butylbenzene	0.0005	0.005	NA	NA
Styrene	0.0005	0.005	6,300	B
Trichloroethene	0.0005	0.005	2.8	B
tert-Butylbenzene	0.0005	0.005	NA	NA
Tetrachloroethene	0.0005	0.005	0.55	B
Toluene	0.0005	0.005	5,680	A
trans-1,2-Dichloroethene	0.0005	0.005	150	B
trans-1,3-Dichloropropene	0.0005	0.005	1.7	B
Trichlorofluoromethane	0.001	0.010	790	B
Vinyl chloride	0.001	0.010	0.06	B
<i>Semivolatile Organic Compounds (SVOCs) by 8270C^a</i>				
1,2,4-Trichlorobenzene	0.0825	0.165	22	B
1,2-Dichlorobenzene	0.0825	0.165	1,900	B
1,3-Dichlorobenzene	0.0825	0.165	NA	NA
1,4-Dichlorobenzene	0.0825	0.165	2.4	B
2,4-Dinitrotoluene	0.0825	0.165	1.6	B
2,6-Dinitrotoluene	0.0825	0.165	61	B
2-Chloronaphthalene	0.0825	0.165	6,300	B
2-Methylnaphthalene	0.0825	0.165	310	B
2-Nitroaniline	0.330	0.825	610	B
3-Nitroaniline	0.330	0.825	24	B
3,3'-Dichlorobenzidine	0.165	0.330	1.1	B
4-Bromophenyl phenyl ether	0.0825	0.165	NA	NA
4-Chloroaniline	0.0825	0.165	2.4	B
4-Chlorophenyl phenyl ether	0.0825	0.165	NA	NA
4-Nitroaniline	0.330	0.825	24	B
Acenaphthylene	0.0825	0.165	NA	NA
Acenaphthene	0.0825	0.165	2,360	A
Anthracene	0.0825	0.165	11,800	A
Benz (a) anthracene	0.0825	0.165	0.42	A
Benzo (a) pyrene	0.0825	0.165	0.042	A
Benzo (k) fluoranthene	0.0825	0.165	4.22	A
Benzo (b) fluoranthene	0.0825	0.165	0.42	A
Benzo (g,h,i) perylene	0.0825	0.165	NA	NA
Benzyl alcohol	0.0825	0.165	6,100	B
Bis (2-chloroethoxy) methane	0.0825	0.165	180	B
Bis (2-chloroethyl) ether	0.0825	0.165	0.21	B
Bis (2-chloroisopropyl) ether	0.0825	0.165	NA	NA

TABLE 4-6

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR SOIL
BALLARD MINE SHOP
(Page 3 of 4)**

Compound	Soil (mg/kg)		Screening Level (mg/kg)	Source ^b
	MDL	RL		
Bis (2-ethylhexyl) phthalate	0.0825	0.165	35	B
Butyl benzylphthalate	0.0825	0.165	260	B
Chrysene ^c	0.0825	0.165	41.9	A
Di-n-butylphthalate	0.0825	0.165	6,100	B
Di-n-octylphthalate	0.0825	0.165	NA	NA
Dibenz (a,h) anthracene ^c	0.0825	0.165	0.015	B
Dibenzofuran	0.0825	0.165	78	B
Diethyl phthalate	0.0825	0.165	49,000	B
Dimethyl phthalate	0.0825	0.165	NA	NA
Fluoranthene	0.0825	0.165	1,570	A
Fluorene	0.0825	0.165	1,570	A
Hexachlorobenzene	0.0825	0.165	0.30	B
Hexachlorobutadiene	0.0825	0.165	6.2	B
Hexachloroethane	0.0825	0.165	35	B
Indeno (1,2,3-cd) pyrene	0.0825	0.165	0.15	B
Isophorone	0.0825	0.165	510	B
n-Nitrosodiphenylamine	0.0825	0.165	99	B
n-Nitrosodi-n-propylamine ^c	0.0825	0.165	0.069	B
Naphthalene	0.0825	0.165	50	A
Nitrobenzene	0.0825	0.165	4.8	B
Phenanthrene	0.0825	0.165	NA	NA
Pyrene	0.0825	0.165	1,700	B
2,4,5-Trichlorophenol	0.0825	0.165	6,100	B
2,4,6-Trichlorophenol	0.0825	0.165	44	B
2,4-Dichlorophenol	0.0825	0.165	180	B
2,4-Dimethylphenol	0.0825	0.165	1,200	B
2,4-Dinitrophenol ^c	0.330	0.825	120	B
2-Chlorophenol	0.0825	0.165	390	B
2-Methylphenol	0.0825	0.165	NA	NA
2-Nitrophenol	0.0825	0.165	NA	NA
4,6-Dinitro-2-methylphenol	0.330	0.825	4.9	B
4-Chloro-3-methylphenol	0.0825	0.165	NA	NA
4-Nitrophenol	0.330	0.825	NA	NA
Benzoic acid	0.330	5.00	240,000	B
Pentachlorophenol	0.330	0.825	0.89	B
Phenol	0.0825	0.165	18,000	B
Polychlorinated Biphenyls (PCBs) by 8082^a				
PCB-1016	0.00825	0.0165	3.9	B
PCB-1221	0.00825	0.0165	0.14	B
PCB-1232	0.00825	0.0165	0.14	B
PCB-1242	0.00825	0.0165	0.22	B
PCB-1248	0.00825	0.0165	0.22	B
PCB-1254	0.00825	0.0165	0.22	B
PCB-1260	0.00825	0.0165	0.22	B

TABLE 4-6

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR SOIL
BALLARD MINE SHOP
(Page 4 of 4)**

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

^b Screening Level Source evaluated using hierarchy below

A State of Idaho Risk Evaluation Manual for Petroleum Releases Table A7-1 Screening Level
Concentrations for Soil Direct Contact, Draft (IDEQ, 2011)

B USEPA RSLs for Chemical Contaminants at Superfund Sites Residential Soil (USEPA, 2010)

^c Screening Level is less than the MDL

mg/kg – milligrams per kilogram

MDL – method detection limit

NA – not available

RL – reporting limit

TABLE 4-7

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR GROUNDWATER
BALLARD MINE SHOP
(Page 1 of 4)**

Compound	Water (µg/L)		Screening Level	Source ^b
	MDL	RL		
<i>Volatile Organic Compounds (VOCs) by 8260B^a</i>				
1,1,1,2-Tetrachloroethane	0.25	5.0	0.52	C
1,1,1-Trichloroethane	0.25	5.0	200	A
1,1,2,2-Tetrachloroethane ^c	0.20	5.0	0.0672	C
1,1,2-Trichloroethane	0.25	5.0	5.0	A
1,1-Dichloroethane	0.125	5.0	2.4	C
1,1-Dichloroethene	0.50	5.0	NA	NA
1,1-Dichloropropene	0.25	5.0	NA	NA
1,2,3-Trichlorobenzene	0.15	5.0	29	C
1,2,3-Trichloropropane ^c	0.50	5.0	0.000724	C
1,2,4-Trichlorobenzene	0.20	5.0	70	A
1,2,4-Trimethylbenzene	0.25	5.0	15	C
1,2-Dichloroethane	0.25	5.0	5.0	A
1,2-Dichlorobenzene	0.125	5.0	370	C
1,2-Dibromo-3-chloropropane ^c	1.0	5.0	0.00032	C
1,2-Dichloropropane	0.20	5.0	5.0	A
1,2-Dibromoethane (EDB) ^c	0.25	5.0	0.0065	C
1,3,5-Trimethylbenzene	0.25	5.0	370	A
1,3-Dichlorobenzene	0.25	5.0	NA	NA
1,3-Dichloropropane	0.20	5.0	730	C
1,4-Dichlorobenzene	0.125	5.0	75	A
2,2-Dichloropropane	0.25	5.0	NA	NA
2-Chlorotoluene	0.125	5.0	NA	NA
4-Chlorotoluene	0.25	5.0	NA	NA
Acetone	2.5	10	22,000	C
Benzene	0.125	5.0	5.0	A
Bromobenzene	0.125	5.0	88	C
Bromochloromethane	0.20	5.0	NA	NA
Bromodichloromethane ^c	0.25	5.0	0.12	C
Bromoform	0.50	5.0	100	A
Bromomethane	0.50	5.0	8.7	C
Carbon tetrachloride	0.25	5.0	5.0	A
Chlorobenzene	0.125	5.0	91	C
Chloroethane	0.50	10	NA	NA
Chloroform	0.125	5.0	0.19	C
Chloromethane	0.50	10	190	C
cis-1,2-Dichloroethene	0.25	5.0	730	C
cis-1,3-Dichloropropene	0.25	5.0	0.43	C
Dichlorodifluoromethane	0.25	10	390	C
Dibromochloromethane ^c	0.25	5.0	0.15	C
Dibromomethane	0.25	5.0	8.2	C
Ethylbenzene	0.25	5.0	700	A
Hexachlorobutadiene	0.25	5.0	0.87	C
Isopropylbenzene	0.25	5.0	NA	NA
m,p-Xylene	0.50	5.0	10,000	A
Methylene chloride	0.25	5.0	4.8	C
Methyl t-butyl ether (MTBE)	0.50	1.0	31	B
MEK (2-Butanone)	2.5	10	7,100	C

TABLE 4-7

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR GROUNDWATER
BALLARD MINE SHOP
(Page 2 of 4)**

Compound	Water (µg/L)		Screening Level	Source ^b
	MDL	RL		
MIBK (4-methyl-2-pentanone)	2.5	10	NA	NA
n-Butylbenzene	0.25	5.0	NA	NA
n-Propylbenzene	0.125	5.0	1,300	C
Naphthalene	0.20	10	210	B
o-Xylene	0.25	5.0	10,000	A
p-Isopropyltoluene	0.25	5.0	NA	NA
sec-Butylbenzene	0.25	5.0	NA	NA
Styrene	0.125	5.0	100	A
Trichloroethene	0.25	5.0	5.0	B
tert-Butylbenzene	0.25	5.0	NA	NA
Tetrachloroethene	0.25	5.0	5.0	A
Toluene	0.25	5.0	1,000	A
trans-1,2-Dichloroethene	0.25	5.0	110	C
trans-1,3-Dichloropropene ^c	0.50	5.0	0.43	C
Trichlorofluoromethane	0.25	10	1,300	C
Vinyl chloride	0.25	10	2.0	A
Semivolatile Organic Compounds (SVOCs) by 8270C^a				
1,2,4-Trichlorobenzene ^c	2.5	5.0	2.3	C
1,2-Dichlorobenzene	2.5	5.0	370	C
1,3-Dichlorobenzene	2.5	5.0	NA	NA
1,4-Dichlorobenzene	2.5	5.0	75	A
2,4-Dinitrotoluene	2.5	5.0	70	A
2,6-Dinitrotoluene ^c	2.5	5.0	0.22	C
2-Chloronaphthalene	2.5	5.0	2,900	C
2-Methylnaphthalene	2.5	5.0	NA	NA
2-Nitroaniline	12.5	25	370	C
3-Nitroaniline	12.5	25	NA	NA
3,3'-Dichlorobenzidine ^c	2.5	5.0	0.15	C
4-Bromophenyl phenyl ether	2.5	5.0	NA	NA
4-Chloroaniline	2.5	5.0	11	C
4-Chlorophenyl phenyl ether	2.5	5.0	NA	NA
4-Nitroaniline	12.5	25	NA	NA
Acenaphthylene	2.5	5.0	NA	NA
Acenaphthene	2.5	5.0	626	B
Anthracene	2.5	5.0	3,130	B
Benz (a) anthracene ^c	2.5	5.0	0.1	B
Benzo (a) pyrene ^c	2.5	5.0	0.2	A
Benzo (k) fluoranthene ^c	2.5	5.0	0.8	B
Benzo (b) fluoranthene ^c	2.5	5.0	0.1	B
Benzo (g,h,i) perylene	2.5	5.0	NA	NA
Benzyl alcohol	2.5	5.0	3,700	C
Bis (2-chloroethoxy) methane	2.5	5.0	110	C
Bis (2-chloroethyl) ether	2.5	5.0	120	C
Bis (2-chloroisopropyl) ether	2.5	5.0	NA	NA
Bis (2-ethylhexyl) phthalate	2.5	5.0	4.8	C
Butyl benzyl phthalate	2.5	5.0	35	C
Chrysene	2.5	5.0	8.0	B
Di-n-butylphthalate	2.5	5.0	NA	NA

TABLE 4-7

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR GROUNDWATER
BALLARD MINE SHOP
(Page 3 of 4)**

Compound	Water (µg/L)		Screening Level	Source ^b
	MDL	RL		
Di-n-octylphthalate	2.5	5.0	NA	NA
Dibenz (a,h) anthracene	2.5	5.0	NA	NA
Dibenzofuran	2.5	5.0	37	C
Diethyl phthalate	2.5	5.0	29,000	C
Dimethyl phthalate	2.5	5.0	NA	NA
Fluoranthene	2.5	5.0	417	B
Fluorene	2.5	5.0	417	B
Hexachlorobenzene ^c	2.5	5.0	0.042	C
Hexachlorobutadiene ^c	2.5	5.0	0.86	C
Hexachloroethane	2.5	5.0	4.8	C
Indeno (1,2,3-cd) pyrene	2.5	5.0	NA	NA
Isophorone	2.5	5.0	71	C
n-Nitrosodiphenylamine	2.5	5.0	14	C
n-Nitrosodi-n-propylamine	2.5	5.0	NA	NA
Naphthalene	2.5	5.0	210	B
Nitrobenzene ^c	2.5	5.0	0.12	C
Phenanthrene	2.5	5.0	NA	NA
Pyrene	2.5	5.0	313	B
2,4,5-Trichlorophenol	2.5	5.0	50	A
2,4,6-Trichlorophenol	2.5	5.0	6.2	C
2,4-Dichlorophenol	2.5	5.0	110	C
2,4-Dimethylphenol	2.5	5.0	730	C
2,4-Dinitrophenol	12.5	25	73	C
2-Chlorophenol	2.5	5.0	180	C
2-Methylphenol	2.5	5.0	NA	NA
2-Nitrophenol	2.5	5.0	NA	NA
4,6-Dinitro-2-methylphenol	12.5	25	NA	NA
4-Chloro-3-methylphenol	2.5	5.0	NA	NA
4-Nitrophenol	12.5	25	NA	NA
Benzoic acid	10	20	150,000	NA
Pentachlorophenol ^c	12.5	25	0.17	C
Phenol	2.5	5.0	1,100	C
Polychlorinated Biphenyls (PCBs) by 8082^a				
PCB-1016	0.25	0.5	0.5	A
PCB-1221	0.25	0.5	0.5	A
PCB-1232	0.25	0.5	0.5	A
PCB-1242	0.25	0.5	0.5	A
PCB-1248	0.25	0.5	0.5	A
PCB-1254	0.25	0.5	0.5	A
PCB-1260	0.25	0.5	0.5	A

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

^b Screening Level Source evaluated using hierarchy below

A State of Idaho Ground Water Quality Rule (IDAPA 58.01.11)

B State of Idaho Risk Evaluation Manual for Petroleum Releases Table A7-1 Screening Level Concentrations for Groundwater Ingestion, Draft (IDEQ, 2011)

C USEPA RSLs for Chemical Contaminants at Superfund Sites Tap Water (USEPA, 2010)

^c Screening Level is less than the MDL

TABLE 4-7

**TARGET COMPOUND LIST, MDLs, RLs, AND HUMAN HEALTH RISK SCREENING LEVELS
FOR GROUNDWATER
BALLARD MINE SHOP
(Page 4 of 4)**

µg/kg – micrograms per kilogram

µg/L – micrograms per liter

MDL – method detection limit

NA – not available

RL – reporting limit

TABLE 4-8

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8260B (VOCs by GC/MS)
(Page 1 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
MS tuning sample	Prior to initial calibration (ICAL) and at the beginning of each 12-hour period	Per Section 7.3.1 and Table 4 of 8260B	Retune instrument then reanalyzing tuning solution.	Per Section II of L/M VOA NFG, except substitute with method acceptance limits.	If criteria are not met, then R
Minimum five-point ICAL for all target compounds	ICAL prior to sample analysis	<p>System performance check compounds (SPCCs) average response factor (RF) ≥ 0.30 for chlorobenzene and 1,1,1,1-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane.</p> <p>RSD for RFs for CCC: VOCs $\leq 30\%$ and one option below</p> <p><i>option 1 linear-</i> RSD for all analytes $\leq 15\%$</p> <p><i>option 2 linear</i> – linear least squares regression $r \geq 0.995$ for each analyte</p> <p><i>option 3 non-linear</i> – COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)</p>	Correct problem and recalibrate	Per Section III of L/M VOA NFG.	Per Table 16 in L/M VOA NFG
Second-Source Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run	All analytes within $\pm 20\%$ of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section IV of L/M VOA NFG.	%R < 80 or >120% = R

TABLE 4-8

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8260B (VOCs by GC/MS)
(Page 2 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Per Section IV of L/M VOA NFG.	Per Table 17 in L/M VOA NFG
Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	NA	Per Section IV of L/M VOA NFG.	Per Table 17 in L/M VOA NFG
Continuing Calibration Verification (CCV)	Daily before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.30 for chlorobenzene and 1,1,1,1-tetrachloroethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. All calibration analytes within $\pm 20\%$ of expected value	Correct problem then repeat CCV. If that fails, then repeat ICAL and reanalyze all samples since last successful CCV.	Per Section III of L/M VOA NFG.	Per Table 16 in L/M VOA NFG
Internal standards verification	Every field sample, standard, and QC sample	RT ± 30 seconds from RT of the midpoint standard in the ICAL; Extracted Ion Current Profile (EICP) area within -50% to +100% of ICAL midpoint standard.	Inspect GC/MS for malfunctions. Reanalyze samples analyzed during instrument malfunction.	Per Section IX of L/M VOA NFG.	Per Table 24 in L/M VOA NFG
Method blank (or preparation blank)	One per analytical batch	No analyte detected $\geq \frac{1}{2}$ RL	Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank. Apply B-flag to all associated positive results for the specific analyte(s) in the preparation batch.	Per Section V of L/M VOA NFG, except use RL instead of CRDL.	Per Table 18 in L/M VOA NFG, except use RL instead of CRDL

TABLE 4-8

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8260B (VOCs by GC/MS)
(Page 3 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
Laboratory Control Sample (LCS) for all analytes	One LCS per analytical batch	Per Table 4-13	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Per Section VII of L/M VOA NFG, except for LCSs and substitute limits specified on Table 4-13 and ≤ 20 RPD limits.	%R > UCL% = J/UJ; < LCL = J detects, R non-detects
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One MS/MSD per every 20 samples per matrix	Per Table 4-13	Correct problem then reanalyze. If still out, address in case narrative	Per Section VII of L/M VOA NFG, except substitute limits specified on Table 4-13 and ≤ 20 RPD limits.	Per Table 22 in L/M VOA NFG, except use limits on Table 4-13 and ≤ 20 RPD limits.
Surrogate spikes	All field and QC samples	Per Table 8 of 8260B and as listed on Table 4-13	Correct problem then reanalyze. If still out, address in case narrative	Per Section VII of L/M VOA NFG, except substitute limits specified on Table 4-13.	Per Table 22 in L/M VOA NFG, except use limits on Table 4-13.
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("J" flag)	Not applicable	Not applicable

^a National Functional Guidelines (NFG) for Organic Data Review (USEPA, 2008).

^b Refer to NFG for detailed evaluation protocols.

CCC – calibration check compound

EICP – extracted ion current profile

GC/MS – gas chromatography/mass spectrometer

ICAL – initial calibration

L/M VOA – low/medium volatile organic analysis

LCL – lower control limit

MDL – method detection limit

NA – not applicable

QC – quality control

RF – response factor

RL – reporting limit

RPD – relative percent difference

RSD – relative standard deviation

RT – retention time

SPCC – system performance check compound

UCL – upper control limit

VOC – volatile organic compound

TABLE 4-9

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8270C (SVOCs by GC/MS)
(Page 1 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
MS tuning sample	Prior to initial calibration (ICAL) and at the beginning of each 12-hour period	Per Section 7.3.1 and Table 3 of 8270C	Retune instrument then reanalyzing tuning solution.	Per Section II of SVOA NFG, except substitute with method acceptance limits.	If criteria are not met, then R
Minimum five-point ICAL for all target compounds	ICAL prior to sample analysis	<p>System performance check compounds (SPCCs) average response factor (RF) ≥ 0.050.</p> <p>RSD for RFs for CCC: SVOCs $\leq 30\%$ and one option below</p> <p><i>option 1 linear-</i> RSD for all analytes $\leq 15\%$</p> <p><i>option 2 linear</i> – linear least squares regression $r \geq 0.995$ for each analyte</p> <p><i>option 3 non-linear</i> – COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)</p>	Correct problem and recalibrate	Per Section III of SVOA NFG.	Per Table 29 in SVOA NFG
Second-Source Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run	All analytes within $\pm 20\%$ of expected value	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section IV of SVOA NFG.	%R < 80 or > 120% = R
Retention time window position establishment for each analyte	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not	NA	Per Section IV of SVOA NFG.	Per Table 30 in SVOA NFG

TABLE 4-9

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8270C (SVOCs by GC/MS)
(Page 2 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
and surrogate		performed, the initial CCV is used.			
Retention time window calculated for each analyte	Each sample	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT	NA	Per Section IV of SVOA NFG.	Per Table 30 in SVOA NFG
Continuing Calibration Verification (CCV)	Daily before sample analysis and every 12 hours of analysis time	SPCCs average RF ≥ 0.050 . All calibration analytes within $\pm 20\%$ of expected value	Correct problem then repeat CCV. If that fails, then repeat ICAL and reanalyze all samples since last successful CCV.	Per Section IV of SVOA NFG.	Per Table 30 in SVOA NFG
Internal standards verification	Every field sample, standard, and QC sample	RT ± 30 seconds from RT of the midpoint standard in the ICAL; Extracted Ion Current Profile (EICP) area within -50% to +100% of ICAL midpoint standard.	Inspect GC/MS for malfunctions. Reanalyze samples analyzed during instrument malfunction.	Per Section X of SVOA NFG.	Per Table 38 in SVOA NFG
Method blank (or preparation blank)	One per preparatory batch	No analyte detected $\geq \frac{1}{2}$ RL	Assess data. Correct problem. If necessary, prep and analyze method blank and all samples processed with the contaminated blank. Apply B-flag to all associated positive results for the specific analyte(s) in the preparation batch.	Per Section V of SVOA NFG, except use RL instead of CRDL.	Per Table 31 in SVOA NFG, except use RL instead of CRDL
Laboratory Control Sample (LCS) for all analytes	One per preparatory batch	Per Table 4-14	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Per Section VII of SVOA NFG, except for LCSs and substitute limits specified on Table 4-14 and \leq	%R > UCL% = J/UJ; < LCL = J detects, R non-detects

TABLE 4-9

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8270C (SVOCs by GC/MS)
(Page 3 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section ^a	Data Validation Qualification ^b
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per preparatory batch per matrix	Per Table 4-14	Correct problem then reanalyze. If still out, address in case narrative	20 RPD limits. Per Section VII of SVOA NFG, except substitute limits specified on Table 4-14 and ≤ 20 RPD limits.	Per Table 36 in SVOA NFG, except use limits on Table 4-14 and ≤ 20 RPD limits.
Surrogate spikes	All field and QC samples	A listed on Table 4-14	Correct problem then reanalyze. If still out, address in case narrative	Per Section VII of SVOA NFG, except substitute limits specified on Table 4-14.	Per Table 36 in SVOA NFG, except use limits on Table 4-14.
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("J" flag)	Not applicable	Not applicable

^a National Functional Guidelines (NFG) for Organic Data Review (USEPA, 2008).

^b Refer to NFG for detailed evaluation protocols.

CCC – calibration check compound

EICP – extracted ion current profile

GC/MS – gas chromatography/mass spectrometer

ICAL – initial calibration

LCL – lower control limit

MDL – method detection limit

NA – not applicable

QC – quality control

RF – response factor

RL – reporting limit

RPD – relative percent difference

RSD – relative standard deviation

RT – retention time

SPCC – system performance check compound

SVOA – semivolatile organic analysis

SVOC – semivolatile organic compound

UCL – upper control limit

TABLE 4-10

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8082 (PCBs by GC/ECD)
(Page 1 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
Minimum five-point ICAL for all target compounds	ICAL prior to sample analysis	One option below <i>option 1 linear</i> - RSD for all analytes $\leq 20\%$ <i>option 2 linear</i> – linear least squares regression $r \geq 0.995$ for each analyte <i>option 3 non-linear</i> – COD ≥ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem and recalibrate.	Per Section II of AOA NFG.	Per Table 63 in AOA NFG
Second-Source Initial Calibration Verification (ICV)	After ICAL, before beginning a sample run	All target analytes within established retention time windows. All target analytes within $\pm 20\%$ of expected value from ICAL	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Per Section III of AOA NFG.	%R < 80 or >120% = R
Retention time (RT) window position establishment for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study	NA	NA	NA
RT window calculated for each analyte	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	Per Section III of AOA NFG.	Per Table 64 in AOA NFG

TABLE 4-10

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8082 (PCBs by GC/ECD)
(Page 2 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section^a	Data Validation Qualification^b
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence	All target analytes within established retention time windows. All target analytes within $\pm 20\%$ of expected value from ICAL	Correct problem then repeat CCV. If that fails, then repeat ICAL and reanalyze all samples since last successful CCV.	Per Section III of AOA NFG.	Per Table 64 in AOA NFG
Method blank (or preparation blank)	One per preparatory batch	No analyte detected $\geq \frac{1}{2}$ RL	Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank. Apply B-flag to all associated positive results for the specific analyte(s) in the preparation batch.	Per Section IV of AOA NFG, except use RL instead of CRDL.	Per Table 65 in AOA NFG, except use RL instead of CRDL
Laboratory Control Sample (LCS) for all analytes	One per preparatory batch	Per Table 4-15	Correct problem then reanalyze. If still out, re-prepare and reanalyze the LCS and all samples in the preparation batch.	Per Section VII of AOA NFG, except for LCSs and substitute limits specified on Table 4-13 and \leq 20 RPD limits.	Per Table 70 in AOA NFG, except use limits on Table 4-15 and \leq 20 RPD limits.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per preparatory batch per matrix	Per Table 4-15	Correct problem then reanalyze. If still out, address in case narrative.	Per Section VI of AOA NFG, except substitute limits specified on Table 4-15 and \leq 20 RPD limits.	Per Table 67 in AOA NFG, except use limits on Table 4-15 and \leq 20 RPD limits.

TABLE 4-10

SUMMARY OF CALIBRATION AND QC PROCEDURES FOR EPA METHOD 8082 (PCBs by GC/ECD)
(Page 3 of 3)

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action/Lab Flagging Criteria	Data Validation Reference Section ^a	Data Validation Qualification ^b
Surrogate spikes	All field and QC samples	TCX within ± 0.05 min and DCB within ± 0.10 min of mean RT determined from ICAL. Recoveries as listed on Table 4-15.	Correct problem then reanalyze. If still out, address in case narrative.	Per Section V of AOA NFG, except substitute limits specified on Table 4-15.	Per Table 66 in AOA NFG, except use limits on Table 4-15.
Compound Identification	All field and QC samples	RTs of surrogates (TCX and DCB) and targets within calculated RT windows (per lines 3 and 4 above). TCX within ± 0.05 min and DCB within ± 0.10 min of mean RT determined from ICAL (per line 9 above). Percent difference (%D) for detected mean concentrations of targets between the two GC columns is within ± 25.0 . Evaluate pattern against standards; assess possibility of interference and degradation.	See corrective action for lines 3, 4, and 9 above. Report target if %D criterion not met, but report QC failure with the result and note in the case narrative.	Per Section X of AOA NFG.	If RT and/or %D criteria not met, flag as U, R, or N per professional judgment as noted in AOA NFG.
Concentrations between the MDL and RL	All samples	Not applicable	Flag as estimated value ("J" flag)	Not applicable	Not applicable

^a National Functional Guidelines (NFG) for Organic Data Review (USEPA, 2008).

^b Refer to NFG for detailed evaluation protocols.

AOA – Aroclors Organic Analysis
 GC/ECD – gas chromatograph/electron capture detector
 ICAL – initial calibration
 LCL – lower control limit
 MDL – method detection limit
 NA – not applicable
 PCB – polychlorinated biphenyl

QC – quality control
 RF – response factor
 RL – reporting limit
 RPD – relative percent difference
 RSD – relative standard deviation
 RT – retention time
 UCL – upper control limit

TABLE 4-11

**VOLATILE ORGANIC COMPOUNDS
RECOVERY LIMITS FOR LABORATORY CONTROL SAMPLES
BALLARD MINE SHOP
(Page 1 of 2)**

Parameter	Analyte	Water (% Recovery)		Soil (% Recovery)	
		LCL	UCL	LCL	UCL
Volatile Organic Compounds (VOCs)	1,1,1,2-Tetrachloroethane	80	130	71	137
	1,1,1-Trichloroethane	80	134	70	135
	1,1,2,2-Tetrachloroethane	79	125	55	130
	1,1,2-Trichloroethane	80	125	60	125
SW8260B	1,1-Dichloroethane	80	125	75	125
	1,1-Dichloroethene	80	132	65	135
	1,1-Dichloropropene	75	130	57	138
	1,2,3-Trichlorobenzene	55	140	60	135
	1,2,3-Trichloropropane	75	125	65	130
	1,2,4-Trichlorobenzene	65	135	65	130
	1,2,4-Trimethylbenzene	80	125	75	132
	1,2-Dichloroethane	80	129	63	133
	1,2-Dichlorobenzene	80	125	70	130
	1,2-Dibromo-3-chloropropane	50	130	40	135
	1,2-Dichloropropane	80	120	72	130
	1,2-Dibromoethane (EDB)	80	129	69	130
	1,3,5-Trimethylbenzene	80	127	74	133
	1,3-Dichlorobenzene	80	120	70	130
	1,3-Dichloropropane	80	120	65	128
	1,4-Dichlorobenzene	80	120	70	130
	2,2-Dichloropropane	80	120	66	135
	2-Chlorotoluene	80	127	63	147
	4-Chlorotoluene	80	126	70	138
	Acetone	40	180	20	160
	Benzene	80	121	70	130
	Bromobenzene	80	120	72	131
	Bromochloromethane	65	130	70	130
	Bromodichloromethane	80	131	72	137
	Bromoform	70	130	49	136
	Bromomethane	30	145	37	143
	Carbon tetrachloride	65	140	59	136
	Chlorobenzene	80	120	70	130
	Chloroethane	60	135	52	135
	Chloroform	80	125	74	129
	Chloromethane	40	125	30	131
	cis-1,2-Dichloroethene	70	125	70	130
	cis-1,3-Dichloropropene	70	130	70	142
	Dichlorodifluoromethane	40	160	25	130
	Dibromochloromethane	60	135	59	136
	Dibromomethane	75	125	69	130
	Ethylbenzene	80	122	70	130
	Hexachlorobutadiene	72	132	65	135
	Isopropylbenzene	80	122	68	129
	m,p-Xylene	80	122	70	130
	Methylene chloride	80	123	74	128
	Methyl t-butyl ether (MTBE)	65	125	54	151
	MEK (2-Butanone)	10	170	37	180
	MIBK (4-methyl-2-pentanone)	64	140	47	146
	n-Butylbenzene	80	131	70	136

TABLE 4-11

**VOLATILE ORGANIC COMPOUNDS
RECOVERY LIMITS FOR LABORATORY CONTROL SAMPLES
BALLARD MINE SHOP
(Page 2 of 2)**

Parameter	Analyte	Water (% Recovery)		Soil (% Recovery)	
		LCL	UCL	LCL	UCL
	n-Propylbenzene	80	129	72	136
	Naphthalene	59	149	50	146
	o-Xylene	80	122	70	130
	p-Isopropyltoluene	80	122	72	128
	sec-Butylbenzene	80	127	71	132
	Styrene	80	123	74	130
	Trichloroethene	80	122	72	126
	tert-Butylbenzene	80	126	72	130
	Tetrachloroethene	80	124	72	130
	Toluene	80	124	77	126
	trans-1,2-Dichloroethene	80	127	72	127
	trans-1,3-Dichloropropene	80	130	65	139
	Trichlorofluoromethane	62	151	48	154
	Vinyl chloride	50	170	45	140
	<i>Surrogate Standards (all samples, standards, and quality control samples)</i>				
	Dibromofluoromethane	86	118	80	120
	1,2-Dichloroethane-d4	80	120	80	120
	Toluene-d8	88	110	81	117
	4-Bromofluorobenzene	86	115	74	121

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

LCL – lower control limit for laboratory control sample

UCL – upper control limit for laboratory control sample

TABLE 4-12

**SEMIVOLATILE ORGANIC COMPOUNDS
RECOVERY LIMITS FOR LABORATORY CONTROL SAMPLES
BALLARD MINE SHOP
(Page 1 of 2)**

Method ^a	Analyte	Water (% Recovery)		Soil (% Recovery)	
		LCL	UCL	LCL	UCL
Semivolatile organic compounds (SVOCs) SW8270C	1,2,4-Trichlorobenzene	25	105	35	100
	1,2-Dichlorobenzene	25	110	35	95
	1,3-Dichlorobenzene	25	110	35	100
	1,4-Dichlorobenzene	25	110	35	105
	2,4-Dinitrotoluene	50	139	50	130
	2,6-Dinitrotoluene	50	120	50	125
	2-Chloronaphthalene	25	120	40	105
	2-Methylnaphthalene	25	120	35	115
	2-Nitroaniline	45	115	45	120
	3-Nitroaniline	40	120	50	130
	3,3'-Dichlorobenzidine	30	140	40	140
	4-Bromophenyl phenyl ether	40	115	40	115
	4-Chloroaniline	25	120	35	100
	4-Chlorophenyl phenyl ether	35	120	40	110
	4-Nitroaniline	53	135	35	140
	Acenaphthylene	30	120	40	110
	Acenaphthene	30	120	40	110
	Anthracene	55	130	55	130
	Benz (a) anthracene	60	130	50	130
	Benzo (a) pyrene	55	135	50	130
	Benzo (k) fluoranthene	55	140	45	135
	Benzo (b) fluoranthene	45	125	45	125
	Benzo (g,h,i) perylene	45	140	40	140
	Benzyl alcohol	20	110	30	100
	Bis (2-chloroethoxy) methane	20	105	30	100
	Bis (2-chloroethyl) ether	25	110	30	100
	Bis (2-chloroisopropyl) ether	20	110	20	115
	Bis (2-ethylhexyl) phthalate	50	150	50	150
	Butyl benzylphthalate	55	150	50	150
	Chrysene	55	130	55	140
	Di-n-butylphthalate	55	118	55	140
	Di-n-octylphthalate	40	146	40	145
	Dibenz (a,h) anthracene	45	125	40	140
	Dibenzofuran	35	115	35	110
	Diethyl phthalate	45	120	50	130
	Dimethyl phthalate	25	112	45	115
	Fluoranthene	50	137	55	140
	Fluorene	40	120	45	115
	Hexachlorobenzene	50	130	45	120
	Hexachlorobutadiene	24	105	30	100
	Hexachloroethane	25	95	30	100
	Indeno (1,2,3-cd) pyrene	50	135	50	135
	Isophorone	30	110	35	100
	n-Nitrosodiphenylamine	40	110	50	130
	n-Nitrosodi-n-propylamine	28	120	35	100
	Naphthalene	25	110	35	100

TABLE 4-12

**SEMIVOLATILE ORGANIC COMPOUNDS
RECOVERY LIMITS FOR LABORATORY CONTROL SAMPLES
BALLARD MINE SHOP
(Page 2 of 2)**

Method ^a	Analyte	Water (% Recovery)		Soil (% Recovery)	
		LCL	UCL	LCL	UCL
	Nitrobenzene	30	110	35	100
	Phenanthrene	55	120	50	130
	Pyrene	55	130	35	140
	2,4,5-Trichlorophenol	35	120	40	110
	2,4,6-Trichlorophenol	30	120	40	110
	2,4-Dichlorophenol	20	110	35	110
	2,4-Dimethylphenol	20	120	30	105
	2,4-Dinitrophenol	20	140	40	130
	2-Chlorophenol	25	110	35	105
	2-Methylphenol	20	110	35	100
	2-Nitrophenol	20	115	35	100
	4,6-Dinitro-2-methylphenol	40	145	45	130
	4-Chloro-3-methylphenol	25	110	40	100
	4-Nitrophenol	10	132	45	140
	Benzoic acid	10	100	20	110
	Pentachlorophenol	40	140	50	150
	Phenol	10	120	35	100
<i>Surrogate Standards (all samples, standards, and quality control samples)</i>					
	2,4,6-Tribromophenol	10	123	19	122
	2-Fluorobiphenyl	43	116	30	115
	2-Fluorophenol	21	100	25	121
	Nitrobenzene-d5	35	114	23	120
	p-Terphenyl-d14	33	141	18	137
	Phenol-d5	10	94	24	113

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

LCL – lower control limit for laboratory control sample

UCL – upper control limit for laboratory control sample

TABLE 4-13

**POLYCHLORINDATED BIPHENYLS
RECOVERY LIMITS FOR LABORATORY CONTROL SAMPLES
BALLARD MINE SHOP**

Parameter	Analyte	Water (% Recovery)		Soil (% Recovery)	
		LCL	UCL	LCL	UCL
Polychlorinated biphenyls (PCBs) SW8082A	PCB-1016	40	140	40	140
	PCB-1221	32	137	64	136
	PCB-1232	32	137	64	136
	PCB-1242	32	137	64	136
	PCB-1248	32	137	64	136
	PCB-1254	60	130	60	130
	PCB-1260	40	140	60	130
	Surrogate Standards (all samples, standards, and quality control samples)				
	2,4,5,6-Tetrachloro-m-xylene	30	132	29	133
	Decachlorobiphenyl	36	144	30	173

^a EPA Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW-846) (USEPA, 1996).

LCL – lower control limit for laboratory control sample

UCL – upper control limit for laboratory control sample

TABLE 4-14

SUMMARY OF LABORATORY INSTRUMENT MAINTENANCE ACTIVITIES

Method/Instrument	Maintenance Type	Description and Frequency
Volatile organic compounds by 8260B using GC/MS	Preventative - systematic	To eliminate the potential for interferences from other areas of the laboratory (e.g., volatilization of solvents), the volatile laboratory shall have an independent air intake system and maintain positive air pressure.
	Preventative	<ul style="list-style-type: none"> When peak shape is deformed: Clip front portion of the analytical column. Daily: Monitor gas pressures; change gas tanks when pressure is below 500 psi. Semiannually: Perform leak tests of entire system.
	Corrective action (as a result of failing calibration or systematic failure of quality control samples)	One or more of the following: replace trap; increase temperatures of entire system (purge and trap apparatus and GC) to clear system of residual contamination; replace column; replace transfer lines; service auto-sampler, sample concentrator, GC, and/or MS (e.g., clean source, replace worn parts).
Semivolatile organic compounds by 8270C using GC/MS	Preventative - systematic	Eliminate potential for interferences from use of specific materials in the laboratory (e.g., materials containing plasticizers).
	Preventative	<ul style="list-style-type: none"> Weekly or as needed: Replace injector ports and septa. When peak shape is deformed: Clip front portion of the analytical column. Daily: Monitor gas pressures; change gas tanks when pressure is below 500 psi. Semiannually: Perform leak tests of entire system.
	Corrective action (as a result of failing calibration or systematic failure of quality control samples)	One or more of the following: replace septa; increase temperatures of entire system (injection port and oven in GC) to clear system of residual contamination; replace column; service auto-sampler, GC, and/or MS (e.g., clean source, replace worn parts).
Polychlorinated biphenyls by 8082 using GC/ECD	Preventative - systematic	Eliminate potential for interferences from use of specific materials in the laboratory (e.g., materials containing plasticizers).
	Preventative	<ul style="list-style-type: none"> Weekly or as needed: Replace injector ports and septa. When peak shape is deformed: Clip front portion of the analytical column. Daily: Monitor gas pressures; change gas tanks when pressure is below 500 psi. Semiannually: Perform leak tests of entire system.
	Corrective action (as a result of failing calibration or systematic failure of quality control samples)	One or more of the following: replace septa; increase temperatures of entire system (injection port and oven in GC) to clear system of residual contamination; replace column; service auto-sampler, GC, and/or ECD (e.g., clean, replace).

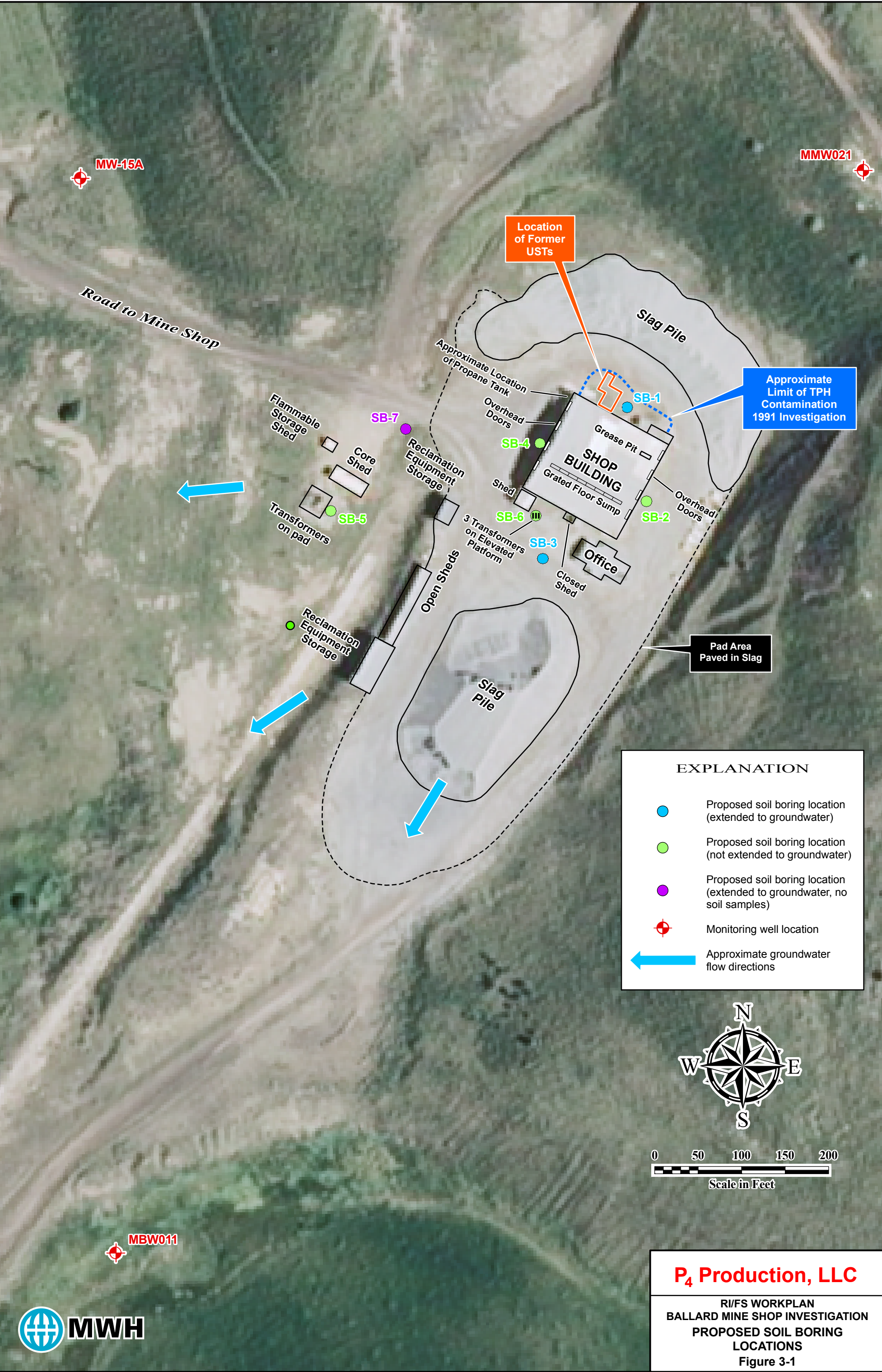
GC/ECD – gas chromatograph/electron capture detector
GC/MS – gas chromatograph/mass spectrometer

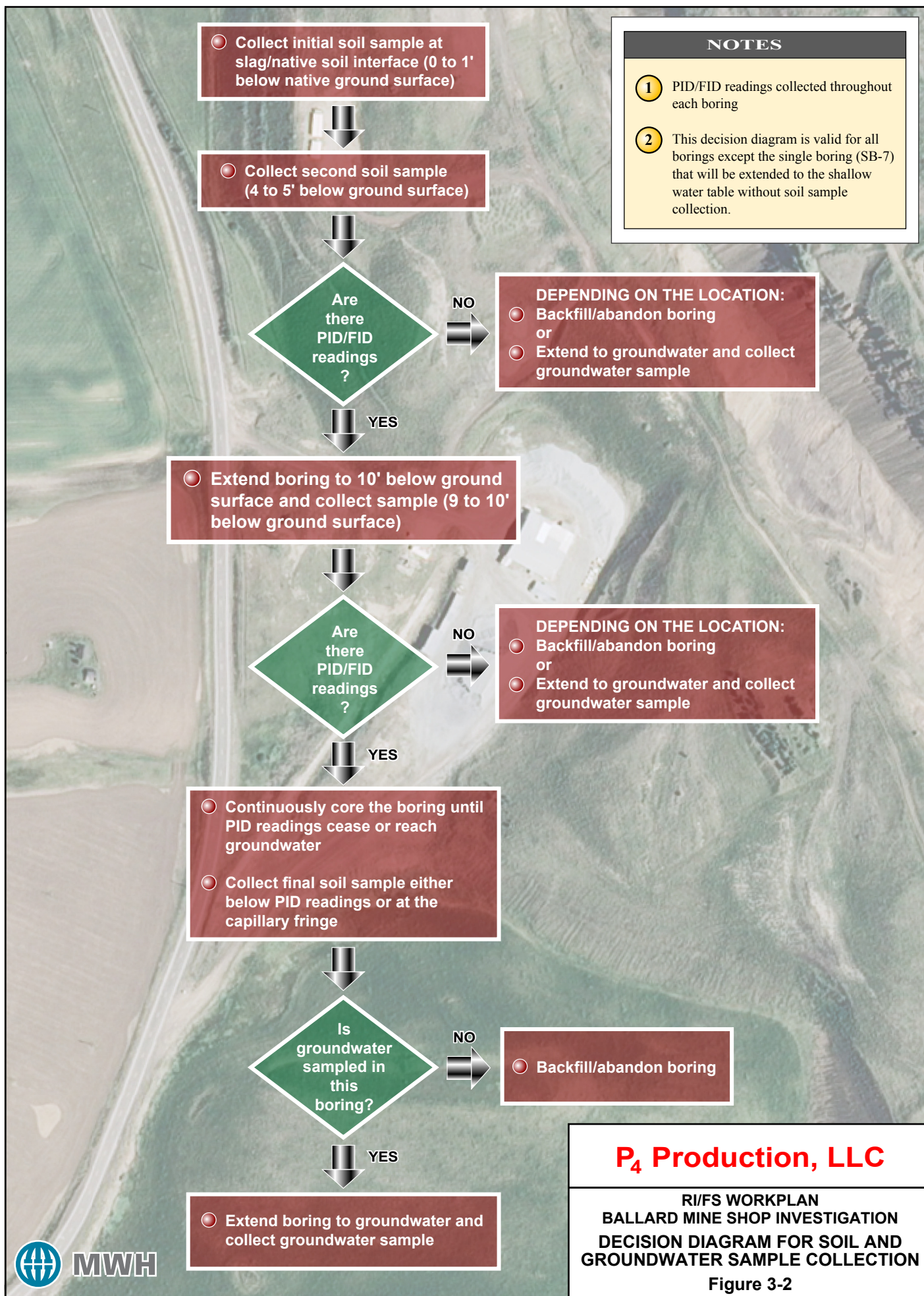
psi – pounds per square inch

**TABLE 5-1
PROJECT CONTACTS**

Company or Agency	Contact	Title	Telephone
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USEPA	Dave Tomten	Remedial Project Manager	208-378-5763
MWH	Vance Drain	MWH Project Manager	801-617-3250
	Ruth Siegmund	Project Chemist	925-627-4756
	Leah Wolf Martin	RI/FS Task Manager	970-871-4364
	Emily Jackson	On-Site Safety Officer	801-617-3232
	Celeste Christensen	Project Coordinator	425-896-6969
Microbac	Kathy Albertson	Project Manager (primary laboratory)	800-373-4071 x179
Laboratory Data Consultants, Inc	Linda Rauto	Project Manager (data validation subcontractor)	760-634-0437

FIGURES





APPENDICES
FOR
FIELD SAMPLING PLAN (FSP)
AND
QUALITY ASSURANCE PROJECT PLAN (QAPP)

APPENDIX A

FSP/QAPP

STANDARD OPERATING PROCEDURES

SOP-1

**BOREHOLE DRILLING, LOGGING, AND
TEMPORARY MONITORING POINT INSTALLATION**

Borehole Drilling, Logging, and Temporary Monitoring Point Installation

Standard Operating Procedures

FEBRUARY 2011

Prepared by:



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LIST OF ATTACHMENTS

<u>Attachment</u>	<u>Description</u>
A	Glossary of Terms
B	Lithologic Logging Form
C	Unified Soil Classification System
D	Soil Boring Log Form
E	Criteria for Describing Plasticity
F	Criteria for Describing Density and Consistency
G	Criteria for Describing Structure
H	Typical Monitoring Well Installation Diagram
I	Monitoring Well Construction Form
J	Water Level Readings Form

1.0 INTRODUCTION

The purpose of this document is to define the standard procedures for drilling, logging, testing, documentation, and installation of temporary monitoring points (TMP). This SOP provides descriptions of equipment, field procedures, and technical procedures necessary to perform the proposed drilling and sampling activity. The procedures described herein are intended to be used with other applicable SOPs, as appropriate.

This SOP describes procedures for conducting the tasks listed below.

- Drilling boreholes
- Sampling soil and bedrock for lithologic description
- Borehole logging
- Equipment decontamination
- TMP design and construction
- TMP sampling

Many terms included in this SOP may be unfamiliar to the reader. A glossary of terms is included in Attachment A.

2.0 DRILLING OPERATIONS

This section provides a description of the principles of operation and the applicability and implementability of the drilling methods that are proposed for this investigation. It focuses on methods and equipment that are readily available and typically applied. It is not intended to provide an all-inclusive discussion of drilling methods. All drillers and drilling personnel working onsite will have the appropriate training (e.g., 40 hour OSHA (CFR 1910) certified and 24 hour MSHA certified). Drillers will also be available to provide additional services for minor repair or servicing of existing wells.

2.1 Hollow-Stem Auger Drilling

Drilling is accomplished by rotating a pipe or rod that has a cutting bit. The common auger drilling method expected to be used is discussed in section.

Hollow-stem augers (HSA) are commonly used in unconsolidated materials up to 150 feet in depth. A key advantage of HSA drilling is that undisturbed soil samples can be collected through the auger, which acts as a temporary outer casing during drilling. The auger also acts as a temporary outer casing during monitoring well installation.

Hollow-stem augers consist of two parts: a tube with flights attached to the outside and connected to the lead auger, and an inner pilot or center rod and bit which is removable from the center of the auger. The removable inner plug is the primary advantage of this drilling method. Withdrawing the plug while leaving the auger in place provides an open, cased hole into which soil samplers, down-hole drive hammers, instruments, casing, wire, pipe, or numerous other items can be inserted. Replacing the center bit and plug allows for continuation of the borehole.

Hollow-stem augers are specified by the inside diameter of the hollow stem, not by the hole size it drills. Hollow-stem augers are available in a variety of diameters, such as 2.5, 3.25, 3.375, 4.0, 4.25, 6.25, 6.625, 8.25, and 10.25 inches. The most commonly used sizes are 3.25 inches and 4.25 inches for soil borings that may be completed as 2-inch monitoring wells, and 6.625 inches for soil borings that may be completed as 4-inch monitoring wells.

The rotation of the augers causes the cuttings to move upward and be "smeared" along the borehole walls. This smearing may effectively seal off the upper zones thereby reducing the possibility of cross contamination of the upper zones to the deeper zones but increases the possibility of deep to shallow contamination. Conversely, smearing of clays on the borehole walls may seal off aquifers to be monitored.

Applications

- Suitable for all types of soil investigations.
- Allows good soil sampling with split-spoon samplers or Shelby tubes.
- Monitoring well installation in all unconsolidated formations.
- Can serve as temporary casing.
- Can be used in stable formations to set surface casing.

Limitations

- Difficulty in preserving sample integrity in heaving formations.
- Formation invasion by water or drilling mud if used to control heaving.
- Possible cross contamination of aquifers where annular space not positively controlled by water or drilling mud or surface casing.
- Limited diameter of augers limits casing size.
- Smearing of clays may seal off aquifer to be monitored.

2.2 Permitting

Temporary monitoring points proposed to be installed will be installed and constructed in accordance with all applicable Idaho Department of Water Resources (IDWR) rules and regulations. For this CERCLA action specific permits do not need to be filed with IDWR, but all well construction needs to be consistent with IDWR rules and regulations.

A licensed drilling subcontractor registered with IDWR will conduct all drilling and well installation activities.

2.3 Lithologic Sampling

A field engineer/geologist will maintain a drill log noting lithology, sampling interval, and other pertinent information. It is anticipated that samples will be collected according to the FSP. More details in lithologic logging can be seen in Section 3.0 and a copy of the lithologic sampling form is presented in the attachments.

2.4 Borehole or Well Abandonment

Any borehole to groundwater that will not be converted into a temporary monitoring point (e.g., soil borings, bedrock boreholes) will be abandoned according to all applicable IDWR rules and regulations. The borehole will be abandoned by pumping cement-bentonite grout to the bottom of

the borehole through a tremie pipe until the borehole is filled to the ground surface with undiluted grout. After 24 hours, the abandoned borehole will be checked for grout settlement. Any settlement will be filled in with grout, using a tremie pipe if it is deeper than 15 feet. This process will be continued until firm grout remains at the ground surface. Shallow boreholes that are not extended to groundwater will be backfilled with the soil removed during drilling and sampling operations.

If the TMPs need to be abandoned, the TMP will be abandoned according to IDWR regulations, and the proper forms will need to be filed with IDWR prior to commencement of abandonment procedures for any permitted well.

2.5 Drilling Equipment Decontamination

All equipment that may directly contact samples for chemical analysis, such as split-spoon samples or core barrels, will be decontaminated on-site. The following sampling-specific decontamination procedures will be utilized.

- Wash and scrub with detergent (laboratory grade, non-phosphate detergent)
- Rinse with potable water
- Rinse with deionized water
- Rinse with another batch of deionized water
- Air dry
- Protect from fugitive dust and vapors

3.0 BOREHOLE LOGGING - SOILS

3.1 General

The procedures described herein are applicable to logging soils and are based on the Unified Soils Classification System (USCS); ASTM Standard D 2488-93, Standard Practice for Description and Identification of Soils (Visual Manual); and ASTM Standard D 5434-93, Standard Guide for Field Logging of Subsurface Explorations of Soil and Rock (ASTM, 1993).

Much of the information described in this section is summarized on several tables and in a USCS field guide, as shown in Attachment C. Other field guidance references also may be used according to personal preference; however, such references must be based on the USCS. Note that many references (for example, AGI Data Sheet grain size scales) are base soil classifications on the Wentworth Scale. Such scales may vary significantly from the USCS and will lead to inaccurate or inconsistent soil descriptions.

All soil logging will be documented using the Lithologic or Soil Boring Log Form included as Attachments B and D, respectively.

3.2 Geologist/Hydrogeologist

One or more geologists or hydrogeologist will accompany each operating drill rig for inspection of drilling and borehole testing work. Each individual will be responsible for only one operating rig. Once assigned to an individual borehole, that person will remain as the geologist or hydrogeologist until that borehole is completed, unless approved for replacement. The geologist or hydrogeologist will be present during the entire time that the drill rig is operating and during casing and screen installation, developing and clean-out operations.

The geologist or hydrogeologist will observe and record the drilling operations along with the characteristics of the subsurface materials. This individual will be responsible for the preparation of a separate log for each boring and will sign each log.

3.3 Definitions

Use of the USCS requires familiarity with the grain size ranges that define a particular type of soil, as well as several other physical characteristics. The grain size definitions and physical characteristics upon which soil descriptions are based are presented below. These procedures are used for soil and other unconsolidated materials.

3.3.1 Grain Sizes

USCS grain sizes are based on U.S. standard sieve sizes, which are listed below.

- Standard sieves with larger openings are named according to the size of the openings in the sieve mesh. For example, a "3-in." sieve contains openings that are 3 inches square.
- Standard sieves with smaller openings are given numbered designations that indicate the number of openings per inch. For example, a "No. 4" sieve contains 4 openings per inch.

The following grain size definitions are paraphrased from the ASTM Standard D 2488-93. Field personnel should familiarize themselves with the grain size definitions.

Boulders - Particles of rock that will not pass a 12-in. (300-mm) square opening.

Cobbles - Particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve.

Gravel - Particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

- *Coarse Gravel* - passes a 3-in. (75-mm) sieve and is retained on a 3/4-in. (19-mm) sieve
- *Fine Gravel* - passes a 3/4-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve

Sand - Particles of rock that will pass a No. 4 (0.19 in. or 4.75-mm) sieve and be retained on a No. 200 (0.003 in. or 75- μ m) sieve with the following subdivisions:

- *Coarse Sand* - passes a No. 4 (0.19 in. or 4.75-mm) sieve and is retained on a No. 10 (0.08 in. or 2-mm) sieve
- *Medium Sand* - passes a No. 10 (0.08 in. or 2-mm) sieve and is retained on a No. 40 (0.017 in. or 425- μ m) sieve
- *Fine Sand* - passes a No. 40 (0.017 in. or 425- μ m) sieve and is retained on a No. 200 (0.003 in. or 75- μ m) sieve

Silt - Soil passing a No. 200 (0.003 in. or 75- μ m) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air-dried. Individual silt particles are not visible to the naked eye.

Clay - Soil passing a No. 200 (0.003 in. or 75- μ m) sieve that can be made to exhibit plasticity within a range of water contents and that exhibits considerable strength when air-dried. Individual clay particles are not visible to the naked eye.

3.3.2 Physical Characteristics

The following physical characteristics are used in the USCS classification for fine-grained soils. A brief definition of each physical characteristic is presented below. A determination of the type of fine-grained soil present in the sample can generally be made on the basis of plasticity, as described in Section 3.4.1.2.

Dry Strength - The ease with which a dry lump of soil crushes between the fingers.

Dilatancy Reaction - The speed with which water appears in a moist pat of soil when shaking in the hand and disappears while squeezing.

Toughness - The strength of a soil, moistened near its plastic limit, when rolled into a 1/8-inch diameter thread.

Plasticity - The extent to which a soil may be rolled into a 1/8-inch thread and re-rolled when drier than the plastic limit.

3.4 Soil Logging Procedures

The following aspects of a project must be understood before sampling and soil logging commences.

- Purpose of the soil logging (e.g., initial investigation, subsequent investigation, remediation)

- Known or anticipated hydrogeologic setting including presence of fill material, lithology, physical characteristics of the aquifer, type of aquifer, recharge/discharge conditions, aquifer thickness and ground water/conditions
- Drilling conditions
- Previous soil boring or borehole geophysical logs
- Soil sampling and geotechnical testing program
- Characteristics of potential chemical release(s) (chemistry, density, viscosity, reactivity and concentration)
- Health and Safety protection requirements
- Regulatory requirements

The procedures used to determine the correct soil sample classification are described below. These procedures are presented in Attachment C through F.

The soils should be described in terms of lithologic units, rather than on a sample-by-sample basis. Thus, a single description may cover several sample intervals, or conversely, several units may occur within a single sample interval. For a specific unit, the primary classification is described and then variations or minor changes are noted below the main description at the depth where they occur.

3.4.1 Field Classification of Soils

When naming soils, the proper USCS soil group name is given followed by the group symbol. For clarity, it is recommended that the group symbol be placed in parentheses after the written soil group name.

Soil identification using the visual-manual procedures is based on naming the portion of the soil sample that will pass a 3-in. (75-mm) sieve. Therefore, before classifying a soil, any particles larger than 3 inches (cobbles and boulders) should be removed, if possible. Estimate and note the percentage of cobbles and boulders.

Using the remaining soil, the next step of the procedure is to estimate the percentages by dry weight of the gravel, sand and fine fractions (particles passing a No. 200 sieve). The percentages shall be estimated to the closest 5%. In general, the soil is *fine-grained* (e.g., a silt or a clay) if it contains 50% or more fines and *coarse-grained* (e.g., a sand or a gravel) if it contains less than 50% fines. If one of the components is present but estimated to be less than 5%, its presence is indicated by the term *trace*. For example, "trace of fines" would be added as additional information following the formal USCS soil description.

3.4.1.1 Procedure for Identifying Coarse-Grained Soils (contain less than 50% fines)

If it has been determined that the soil contains less than 50% fines, the soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand. The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

If the soil is predominantly sand or gravel but contains an estimated 15% or more of the other coarse-grained constituent, the words "with gravel" or "with sand" shall be added to the group name. For example: "gravel with sand (GP)." If the sample contains any cobbles or boulders, the words "with cobbles" or "with cobbles and boulders" shall be added to group name. For example: "silty gravel with cobbles (GM)."

5% or less fines. The soil is a "clean gravel" or "clean sand" if the percentage of fines is estimated to be 5% or less. "Clean" is not a formal USCS name but rather a general descriptor for implying little to no fines. Clean sands and gravels are given the USCS designation as either *well-graded* or *poorly-graded*, as described below.

Identify the soil as a *well-graded gravel* (GW) or as a *well-graded sand* (SW), if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes. Identify the soil as a *poorly-graded gravel* (GP) or as a *poorly-graded sand* (SP) if it consists predominantly of one grain size (uniformly graded) or has a wide range of sizes with some intermediate sizes obviously missing (gap- or skip-graded).

Note: When using the USCS, keep in mind the difference between grading and sorting. The term grading is used to indicate the range of particles contained in the sample. For example, a poorly-graded sand containing predominantly one grain size would be considered well-sorted and vice-versa. One notable exception to this general rule is a skip-graded (bimodally distributed) sample: a sand containing two distinct grain sizes would be considered both poorly-sorted and poorly-graded. The USCS uses only the *GRADING* descriptor in soil naming, not the sorting descriptor.

≥ 15% fines. The soil is a *silty* or *clayey gravel* or a *silty* or *clayey sand* if the percentage of fines is estimated to be 15% or more. For example, identify the soil as *clayey gravel* (GC) or a *clayey sand* (SC) if the fines are clayey. Identify the soil as a *silty gravel* (GM) or a *silty sand* (SM) if the fines are silty. The coarse-grained descriptor "poorly-graded" or "well-graded" is not included in the soil name, but rather, should be included as additional information following the formal USCS soil description.

>5% but <15% fines. If the soil is estimated to contain greater than 5% and less than 15% fines, give the soil a dual identification using two group symbols. The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a clayey/silty gravel or sand (GC, GM, SC, SM). The group name shall correspond to the first group symbol and include the words "poorly-graded" or "well-graded", plus the words "with clay" or "with silt" to indicate the character of the fines. For example, "poorly-graded gravel with silt (GP-GM)".

3.4.1.2 Procedure for Identifying Fine-Grained Soils (contain 50% or more fines)

The USCS classifies inorganic fine-grained soils according to their degree of plasticity (no or low plasticity - indicated with an "L", or high plasticity - indicated with an "H"). The field tests used to determine dry strength, dilatancy and toughness are generally too time consuming to be performed on a routine basis. Field personnel should be familiar with the definitions of the physical characteristics and the concepts of the field tests; however, field classifications will generally be based primarily on plasticity, as described in Attachment E.

Lean clay (CL) - soil has medium to high dry strength, no or slow dilatancy and medium toughness and plasticity.

Fat clay (CH) - soil has high to very high dry strength, no dilatancy and high toughness and plasticity.

Silt (ML) - the soil has no to low dry strength, slow to rapid dilatancy and low toughness and plasticity, or is nonplastic.

Elastic silt (MH) - the soil has low to medium dry strength, no to slow dilatancy and low to medium toughness and plasticity; will air dry more quickly than lean clay and have a smooth, silky feel when dry.

Organic soil (OL or OH) - the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, from black to brown, when exposed to the air. Organic soils normally will not have a high toughness or plasticity.

Other Modifiers for Use with Fine-Grained Soils:

15% to 25% coarse-grained material. If the soil is estimated to have 15% to 25% sand or gravel, or both, the words "with sand" or "with gravel" (whichever is predominant) shall be added to the group name. For example: "lean clay with sand (CL)" or "silt with gravel (ML)." If the percentage of sand is equal to the percentage of gravel, use "with sand."

≥30% coarse-grained material. If the soil is estimated to have 30% or more sand or gravel, or both, the words "sandy" or "gravelly" shall be added to the group name. Add the word "sandy" if there appears to be the same or more sand than gravel. Add the word "gravelly" if there appears to be more gravel than sand. For example: "sandy silt (ML)", or "gravelly fat clay (CH)."

3.4.1.3 Procedure for Identifying Borderline Soils

To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example, a soil containing an estimated 50% silt and 50% fine grained sand may be assigned a borderline symbol "SM/ML." Borderline symbols should not be used indiscriminately. Every effort should be made to first place the soil into a single group and then to estimate percentages following the USCS soil description.

3.4.2 Descriptive Information for Soils

After the soil name and symbol are assigned, the soil color, consistency/density and moisture content shall be described in that order. Other information is presented later in the description, as applicable.

3.4.2.1 Color

Describe the color using the Munsell Soil Color Chart (1992). Color is an important property in identifying organic soils and may also be useful in identifying materials of similar geologic or depositional origin in a given location.

When using the Munsell Soil Color Charts, first attempt to assign the soil a general color, such as brown, gray, red, etc. Then go to the correct area in the charts and assign the applicable color name and Munsell symbol. The ability to detect minor color differences varies among people and the chance of finding a perfect color match in the charts is rare. Keeping this in mind should help field personnel avoid spending unnecessary time and confusion going through the chart pages. In addition, attempting to describe detail beyond the reasonable accuracy of field observations could lead to making poorer soil descriptions than by expressing the dominant colors simply (Munsell Soil Color Chart, 1992).

If the color charts are not being used or are unavailable, again attempt to assign general colors to soils. Comparing a particular soil sample to samples from different locations in the borehole will help keep the eye "calibrated." For example, by holding two soils together, it may become evident that one is obviously greenish-brown, while another is reddish.

3.4.2.2 Consistency & Density

For intact fine-grained soil, describe consistency as very soft, soft, medium stiff, stiff, very stiff, or hard, based on the blows per foot using a 140-pound hammer dropped 30", as described in Attachment F. If blow counts are not available, use the thumb test, as described in Attachment F to determine consistency.

For coarse-grained soils, describe density based on blows per foot as very loose, loose, medium dense, dense and very dense, as described in Attachment F. If blow counts are not available, attempt to estimate the soil density by observation, since a practical field test is not available. Be sure to clearly indicate on the field boring log if blow counts could not be obtained.

3.4.2.3 Moisture

Describe the moisture condition of the soil as dry (absence of moisture, dusty, dry to the touch), moist (damp but no visible water, even in interstices) or wet (visible free water, saturated).

3.4.2.4 Maximum Grain Size

Describe the maximum particle size found in the sample in accordance with the information listed below.

- **Sand Size** - If the maximum particle size is a sand size, describe as fine, medium, or coarse.
- **Gravel Size** - If the maximum particle size is a gravel size, describe the diameter of the maximum particle size in inches.
- **Cobble or Boulder Size** - If the maximum particle size is a cobble or boulder size, describe the maximum dimension of the largest particle.

For gravel and sand components, describe the range of particle sizes within each component. For example, "about 20% fine to coarse gravel, about 40% fine to coarse sand."

3.4.2.5 Odor

Due to health and safety concerns, NEVER intentionally smell the soil. This could result in exposure to volatile contaminants that may be present in the soil. If, however, an odor is incidentally noticed, it should be described if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation (sometimes a hydrogen sulfide ["rotten egg"] smell). If the odor is unusual (petroleum product, chemical, etc.), it should be described. Organic vapor readings from an OVM or similar instrument should be noted on the field boring log. The project-specific Health and Safety Plan should then be consulted to determine the appropriate level of protection necessary for the continuation of fieldwork.

3.4.2.6 Cementation

Describe the cementation of intact coarse-grained soils as weak, moderate or strong, in accordance with the criteria listed below.

- **Weak** - Crumbles or breaks with handling or little finger pressure
- **Moderate** - Crumbles or breaks with considerable finger pressure
- **Strong** - Will not crumble or break with finger pressure

The presence of calcium carbonate may be confirmed on the basis of effervescence with dilute hydrochloric acid, HCl, if calcium carbonate or caliche is believed to be present in the soil. Proper health and safety precautions must be followed when mixing, handling, storing, or transporting HCl.

3.4.2.7 Angularity

Describe the angularity of the sand (coarse sizes only), gravel, cobbles and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria listed below.

- **Angular** - Particles have sharp edges and relatively planar sides with unpolished surfaces
- **Subangular** - Particles are similar to angular description but have rounded edges
- **Subrounded** - Particles have nearly plane sides but have well-rounded corners and edges
- **Rounded** - Particles have smoothly curved sides and no edges

A range of angularity may be stated, such as "subrounded to rounded".

3.4.2.8 Structure

Describe the structure of intact soils in accordance with the criteria in Attachment G.

3.4.2.9 Lithology

Describe the primary lithologies (rock or mineral type) of the sand, gravel, cobbles and boulders, if possible. It may be difficult to determine the lithology of fine and medium-grained sand or particles that have undergone alteration.

3.4.2.10 Additional Comments

Additional comments may include the presence of roots or other vegetation, fossils or organic debris, staining, mottling, or oxidation; difficulty in drilling and caving or sloughing of the borehole walls. Also, when drilling in an area known or suspected to contain imported fill material, every effort should be made to identify the contact between fill and native soils. If a soil is suspected to be fill, this should be clearly indicated on the log following the soil description. Stratigraphic units and their contacts should be noted wherever possible.

3.4.3 Additional Boring Log Information

In addition to soil descriptions, there are several other items that should be included on all *soil boring log forms*, included in Attachment F. Information in the log heading should be complete and accurate. The information listed below should be included, at a minimum.

- Boring or monitoring well number
- Project name and job number
- Site name
- Name of individual who logged the boring
- Drilling contractor
- Drill rig type and method of drilling (for example, "CME 75, hollow stem auger")
- Name of drilling company
- Name of driller and helper
- Borehole diameter and drill bit type
- Type of soil sampler (for example, Modified California, continuous core, etc.)
- Time and date that drilling started and finished
- Time and date that the well was completed or the soil boring backfilled, as appropriate
- Method of borehole abandonment, if applicable
- Sketch map of boring or well location with estimated distances to major site features such as property lines or buildings and north arrow

Soil sample information should include the depth interval that was sampled, the blow counts per six inches, the amount of soil recovered and the portion submitted for analysis or testing, if any. The sample identification number may also be noted on the log.

The degree to which soil samples are collected during a field effort depends on the overall scope and purpose of the investigation, which should be clearly defined before the field effort commences. Additional soil samples may need to be collected if, for example, soils are very heterogeneous or unexpected conditions such as perched water zones or zones of contamination are encountered.

If groundwater is encountered during drilling, the depth to water and the time and date of the observation should be recorded. If the first water encountered is a perched zone, the depth, time

and date that any additional groundwater zones are encountered should also be recorded. Depth to water after drilling, the measuring point and the date and time of the measurement(s) must be noted. Additional measurements of depth to groundwater, including depth and time, may be beneficial.

4.0 TEMPORARY MONITORING POINT AND WELL DESIGN AND INSTALLATION

4.1 General

This guideline is applicable to the design and installation of TMPs and monitoring wells. Each TMP or monitoring wells will be designed to suit the hydrogeologic setting of the site, the type of contaminants to be monitored, the overall purpose of the monitoring program and other site-specific variables. During all phases of TMP design, attention must be given to clear documentation of the basis for design decisions, the details of well construction and the materials to be used. A Typical Monitoring Well Installation Diagram is provided as Attachment H and a Monitoring Well Construction Form is provided in Attachment I.

4.2 TMP Locations

The current scope of work entails installing TMPs that could be converted to monitoring wells in the future. The locations and rationale of these wells are discussed in the SAP and FSP.

4.3 TMP Design

4.3.1 Casing Diameter and Screen Length

TMP casing diameter is dependent on the purpose of the well and the amount and size of downhole equipment that must be accommodated. All of the wells are designed to be TMPs. Therefore, they will all be constructed with 2-inch diameter PVC well casing.

The lengths will be 10 to 20 feet. Any TMPs that will be screened near the water table will be screened across the water table. Consideration should be given to seasonal fluctuations in water levels when locating the well screen across the top of the water table.

4.3.2 Casing and Screen Materials

The two most commonly used materials are PVC and stainless steel. PVC is inexpensive, widely available, lightweight and easy to work with. Many studies have been conducted concerning the effect of PVC on water quality data. Adsorption of some chlorinated species to PVC was found to be too slow to effect data quality. Because a sample is generally taken shortly after the purging of stagnant water in contact with the casing, the contaminants in the water will have minimal time to be influenced by sorption or leaching effects. Therefore, potential sample bias effects due to interactions with PVC are negligible (Reynolds, et al, 1990). Consequently, TMP casings and

screens will be constructed of polyvinyl chloride (PVC). As these TMPs are less than 50 feet deep they will be constructed of schedule 40 PVC.

The hydraulic efficiency of a well screen depends primarily upon the amount of open area available per unit length of screen. The two screen types commonly used for monitoring wells are machine-slotted and continuous-slot wire-wound. The continuous-slot, wire-wound screen has a greater area per opening per length and diameter than is available with any other screen type. The percentage of open area in continuous-slot screen is often more than twice that provided by standard slotted well screen. The triangular shaped wire makes these screens non-clogging. The TMPs installed at the site will be constructed with machine-slotted PVC screens.

Additional construction specifications are listed below.

- Threaded, flush-joint casing
- Well caps that are vented to prevent the accumulation of gases and to allow water levels in the well to respond to barometric and hydraulic pressure changes
- Threaded end-caps

4.3.3 Decontamination of Casing and Screen Materials

During the production of PVC casing, a wax layer can develop on the inner wall of the casing; protective coatings may also be added to enhance casing durability. All of these represent potential sources of chemical interference and must be removed with either a laboratory-grade non-phosphate solution or by steam cleaning prior to installation. Factory cleaning of casing and screen in a controlled environment by standard detergent washing, rinsing and air-drying procedures is superior to any cleaning efforts attempted in the field. Factory cleaned and sealed casing and screen that is certified by the supplier will be used if available.

4.3.4 Filter Pack and Well Screen Design

A properly designed TMP requires that a well screen be placed opposite the zone to be monitored and be surrounded by materials that are coarser and of greater hydraulic conductivity than the natural formation material. Filter packs are installed to create a permeable envelope around the well screen. The selection of the filter pack grain size should be based on the grain size of the finest layer to be screened.

The typical well construction for a monitoring well in average formation materials includes filter pack on the order of #3 Monterey sand size and 0.020 inch slotted screen. For finer formations, 0.010 inch slotted screen may be used with appropriately graded sand (e.g., 20/40). A configuration similar to this will be used, unless the materials encountered are radically different than expected.

If conditions warrant, filter pack grain size and well screen slot size should be determined by the grain size distribution of the formation material. The filter pack should be designed first. It is recommended to use a filter pack grain size that is three to five times the average (D50) size of the formation materials. D50 will be estimated based on the lithologic description made by the site geologist or hydrogeologist. However, this method may be misleading in coarse, well graded formation materials. Another way to determine filter pack grain size is to take the D30 grain size of the formation materials and multiplying it by a factor of between 3 and 6, with 3 used if the formation is fine and uniform and 6 used if the formation is coarse and non-uniform. For both methods, the uniformity coefficient of the filter pack materials should be as close to 1.0 as possible to minimize particle size segregation during filter pack installation.

The filter pack will extend from the bottom of the well screen to approximately 3 to 5 feet above the top of the screen to account for settlement of the pack material during development and to act as a buffer between the well screen and the annular seal. Filter pack thickness must be sufficient to surround the well screen but thin enough to minimize resistance to the flow of fine-grained formation material and water into the well during development. Consequently, a filter pack thickness of approximately 2 inches will be used.

The materials comprising the filter pack should be as chemically inert as possible. It should be comprised of clean quartz sand or glass beads. Filter pack materials usually come in 100-pound bags; these materials are washed, dried and factory packaged.

The size of well intake openings can only be selected after the filter-pack grain size is specified. The slot size should be such that 90 percent to 100 percent of the filter-pack material is held back by the well screen.

The casing string should be installed in the center of the borehole. This will allow the filter-pack materials to evenly fill the annular space around the screen and ensure that annular seal materials fill the annular space evenly around the casing. Where a dual-tube rig is used, the inner tube of the dual tube will adequately centralize the casing string. For other types of drilling, centralizers will be used to ensure the casing string is positioned in the center of the borehole. Centralizers are typically expandable metal or plastic that attach to the outside of the casing and are adjustable along the length of the casing. Centralizers will be attached immediately above the well screen and at 20 to 50-foot intervals along the casing to the surface.

Methods for filter pack emplacement normally used for monitoring wells include: 1) gravity (free-fall); and 2) tremie pipe. Gravity emplacement is only possible in relatively shallow wells (less than

~50 feet) with an annular space of more than 2 inches where the potential occurrence of bridging is minimized. Bridging can result in the occurrence of large unfilled voids in the filter pack or the failure of filter pack materials to reach their intended depth. Gravity emplacement may also cause filter pack gradation. Additionally, formation materials from the borehole wall can become incorporated into the filter pack, potentially contaminating it.

With the tremie emplacement method, the filter pack is poured or slurried into the annular space adjacent to the well screen through a rigid pipe, usually 1.5 inches in diameter. Initially the pipe is positioned so that its end is at the bottom of the annulus. If the filter pack is being installed in a temporarily cased borehole (e.g., dual-tube percussion) the temporary casing is pulled to expose the screen as the filter-pack material builds up around the well screen. In unconsolidated formations the temporary casing should only be pulled out 1 to 2 feet at a time to prevent caving. In consolidated or well-cemented formations or in cohesive unconsolidated formations, the temporary casing may be raised well above the bottom of the borehole prior to filter pack emplacement. For deep wells and/or nonuniform filter pack materials, the filter pack may be pressure fed through a tremie pipe with a pump. Emplacement will be continuously monitored with a weighted measuring tape accurate to the nearest 0.1 foot to determine when the filter pack has reached the desired height.

4.3.5 Annular Seal

Proper annular seal formulation and placement results in the complete filling of the annular space and envelopes the entire length of the well casing to ensure that no vertical migration can occur within the borehole.

Annular seal materials will include bentonite chips or a high solids (approximately 10%) bentonite grout with a weight in the range of eleven to thirteen pounds per gallon of sealant. The grout will be mixed using the manufacturer's directions. A bentonite seal at least 2 feet thick will be emplaced immediately above the filter pack using a side-discharge tremie pipe. The use of bentonite as a sealing material depends on its efficient hydration following emplacement. Expansion of bentonite in water can be on the order of 8 to 10 times the volume of dry bentonite. This expansion causes the bentonite to provide a tight seal between the casing and the adjacent formation. Bentonite pellets, granules, or chip will be used for this seal. Bentonite pellets expand in water at relatively slow rates, thus reducing the potential for bridging compared to chips, chunks, or granules. If the bentonite seal will be above the saturated zone, several gallons of clean distilled water will be poured down the annulus to begin the hydration process. A minimum of 30 minutes should pass to allow for hydration before additional annular seal materials are placed above the bentonite.

The high solids grout will be mechanically blended in an aboveground rigid container and pumped through a tremie pipe to within a few inches of the bottom of the space to be sealed. This allows the grout to displace groundwater and loose formation materials up the hole. The end of the tremie

pipe should always remain in the grout without allowing air spaces. After emplacement, the tremie pipe should be removed immediately. The grout should be emplaced in one continuous mass before initial setting of the cement or before the mixture loses its fluidity.

Cement is a highly alkaline substance (pH from 10 to 12) and introduces the possibility of altering the chemistry of the water it contacts. Thinner slurries may infiltrate an unprotected filter pack. After a borehole annulus is filled with grout a sample of water may be obtained and the pH determined in the field. A pH reading of 12 or higher may indicate an invasion of cement grout into the well.

4.3.6 Surface Completions

In the event that the TMPs are converted to monitoring wells in the future, two types of surface completions will be used: aboveground and flush-mounted. Aboveground completions will be used wherever practical. Flush mounted completions will be used anywhere there may be vehicle traffic or where low visibility is preferred. The primary purpose of either type of completion is to prevent surface runoff from entering and infiltrating down the annulus of the well and to protect the well from accidental damage or vandalism. The surface seal may be an extension of the annular seal installed above the filter pack, or a separate seal emplaced atop the annular seal.

For aboveground completions, a protective steel casing fitted with a locking cover will be set into the uncured cement surface seal. Three to four guard posts (bollards) will be spaced around each well with above ground completions to afford additional protection.

In a flush-mount surface completion, a water-tight monitoring well Christy box or its equivalent will be set into the cement surface seal before it has cured. This type of completion is used in high-traffic areas. A low, gently sloping mound of cement will discourage surface runoff. A locking well cap will be used to secure the inner well casing.

4.3.7 Summary of TMP Design

In summary, the filter pack and TMP design criteria for the investigations are listed below.

- PVC screen and casing
- Schedule 40 casing
- 0.010 or 0.020-inch machine slotted screen
- 2-inch diameter casing

- Threaded flush joint casing and end-caps
- Sand appropriately graded for the well screen for filter packs up to 3 to 5 feet above the top of the screened interval
- Bentonite plug at least 2 feet thick on top of filter pack
- Annular seal to the surface to consist of bentonite or neat cement
- Both filter pack and annular seal are to be emplaced using a tremie pipe

5.0 References

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ATTACHMENTS

ATTACHMENT A

GLOSSARY OF TERMS

Absorption - The penetration or apparent disappearance of molecules or ions of one or more substances into the interior of a solid or liquid.

Adsorption - The process by which atoms, ions, or molecules are held to the surface of a material through ion-exchange processes.

Annular Sealant - Material used to provide a positive seal between the borehole and the casing of the well. Annular sealants should be impermeable and resistant to chemical or physical deterioration.

Annular Space - The space between the borehole wall and the well casing, or the space between a casing pipe and a liner pipe.

Annulus - The gap between the well and borehole where the sand, seal and grout are installed.

Aquifer - A geologic formation, group of formations, or part of a formation that can yield water to a well or a spring.

Backwashing - A method of filter pack emplacement whereby the filter pack material is allowed to fall freely through the annulus while clean fresh water is simultaneously pumped down the casing.

Bentonite - Hydrous aluminum silicate available in powder, granular, or pellet form. It is used to provide a tight seal between the well casing and the borehole.

Bailer - A cylindrical tool designed to remove material, both solid and liquid, from a well or borehole. A valve at the bottom of the bailer retains the material in the bailer. The three types of bailers are flat-valve bailer, a dart-valve bailer and the sand pump with rod plunger.

Blow Counts - Number of hammer blows needed to advance a split spoon sampler. Blow counts are usually counted in 6-inch increments.

Borehole - The hole created by drilling through the subsurface.

Bridge - A wedge or build up of sand that occurs when the driller is pouring the sand pack around the screened interval, thus leaving a gap or "open zone" where the natural formation could possibly clog the screen. Also the development of gaps or obstructions in either grout or filter pack materials during emplacement.

Cone Penetrometer - An instrument used to identify the underground conditions by measuring the differences in the resistance and other physical parameters of the strata. The cone penetrometer consists of a conical point attached to a drive rod of smaller diameter. Penetration of the cone into the formation forces the soil aside, creating a complex shear failure. The cone penetrometer is very sensitive to small differences in soil consistency.

Continuous Slot Wire-Wound Intake - A well intake that is made by winding and welding triangular-shaped, cold-rolled wire around a cylindrical array of rods. The spacing of each successive turn of wire determines the slot size of the intake.

Core Barrel - A steel tube used to collect rock core samples. The core barrel receives the rock core cut by the outer barrel as the borehole is advanced.

Cuttings - Formation particles obtained from a borehole during the drilling process.

Drill Rod - The rigid steel rod used to lower and retrieve cutting, coring and sampling equipment down the borehole.

Draw down - Distance between the static water level and water level while the well is being pumped or bailed at a constant rate.

Drilling Fluids - A water-based or air-based fluid used in the well drilling operation to remove cuttings from the borehole, to clean and cool the bit, to reduce friction between the inner barrel and the sides of the borehole and to seal the borehole.

Dual-Purpose Well - A well that can be used as both a monitoring and extraction or injection well.

Filter Pack - Sand, gravel, or glass beads that are uniform, clean and well-rounded that are placed in the annulus of the well between the borehole wall and the well intake to prevent formation material from entering through the well intake and to stabilize the adjacent formation.

Fines - Silt, clay, fine sand.

Grout - A fluid mixture of neat cement and water with various additives or bentonite of a consistency that can be forced through a pipe and emplaced in the annular space between the borehole and the casing to form an impermeable seal.

Heaving Formation - Unconsolidated saturated substrate encountered during drilling where the hydrostatic pressure of the formation is greater than the borehole pressure causing the sands to move up into the borehole.

Inner Barrel - The tool lowered through the inside of the outer barrel that can be configured for cutting, coring, or sampling.

Kelly Bar - A hollow steel bar or pipe that is the main section of drill string to which the power is directly transmitted from the rotary table to rotate the drill pipe and bit. The cross section of the kelly is either square, hexagonal, or grooved. The kelly works up and down through drive bushings in the rotary table.

Neat Cement - A mixture of Portland cement and water in the proportion of 5 to 6 gallons of clean water per bag (94 pounds) of cement.

Outer Barrel - The steel piping that serves to both cut downwards and to line the borehole walls to prevent hole collapse.

Overshot Tool - The tool that attaches to the inner barrel so that the barrel may be lowered through the outer barrel to depth on the wireline. The overshot tool is designed to attach to, or release from, the inner tube at depth.

Parameters - Groundwater variables, pH, specific conductivity, temperature, turbidity.

Pitch - The distance along the axis of an auger flight that it takes for the helix to make one complete 360 degree turn.

Purge water - Any water removed from the well via bailing, pumping, or air lift.

Rotary Table - A mechanical or hydraulic assembly that transmits rotational torque to the kelly, which is connected to the drill pipe and the bit. The rotary table has a hole in the center through which the kelly passes.

Saturated annulus - The portion of the annulus that is below the aquifer.

Sieve Analysis - Determination of the particle-size distribution of soil, sediment, or rock by measuring the percentage of the particles that will pass through standard sieves of various sizes.

Split-Spoon Sampler - A thick-walled steel tube split lengthwise used to collect soil samples. The sampler is commonly lined with metal sample sleeves and is driven or pushed downhole by the drill rig to collect samples.

Thin-Walled Sampler - A sampling device used to obtain undisturbed soil samples made from thin-wall tubing. The sampler is also known as a Shelby tube. The thin-wall sampler minimizes the most serious sources of disturbance: displacement and friction.

Tremie Pipe - A device, usually a small-diameter pipe, that carries grouting materials to the bottom of the borehole and that allows pressure grouting from the bottom up without introduction of appreciable air pockets.

VOCs - Volatile organic compounds.

Wireline - The steel cable used to lower and retrieve cutting, coring and sampling equipment down the borehole.

Yield - The rate at which a well will produce water.

ATTACHMENT B
LITHOLOGIC LOGGING FORM

PAGE ____ OF ____

BORING LOCATION:

ABBREVIATIONS:		CALC. CONTENT	HYD. PRESS.
DRILLING ACTION		N: Non-calc.	N: None
E: Even, smooth	r: Slightly rough	S: Slightly	L: Low
C: Crunchy	R: Moderately rough	M: Moderately	M: Medium
I: Intermittently rough	<u>R</u> : Very rough	V: Very	F: Full
		Bore by Cut, Page 2, MAC/CAD, Rev: 9-8-95	



MWH

ATTACHMENT C
UNIFIED SOIL CLASSIFICATION SYSTEM

FIELD GUIDE

ORDER OF DESCRIPTION

1. Soil type
2. USCS symbol
3. Color
4. Consistency/Density
5. Moisture
6. Grain size (sands and gravels)
7. Cementation
8. Plasticity
9. Miscellaneous

EXAMPLE DESCRIPTION

Poorly-graded sand with gravel (SP), light brown, loose, moist, predominantly fine sand, trace medium sand, 20% fine gravel, hydro carbon odor and staining

UNIFIED SOIL CLASSIFICATION SYSTEM

1	COARSE-GRAINED SOILS <50% passes #200 sieve	GRAVELS <50% coarse fraction passes #4 sieve	GRAVELS with little or no fines	Well-graded gravels, gravel-sand mixtures, little or no fines	GW	2
				Poorly-graded gravels, gravel-sand mixtures, little or no fines	GP	
			GRAVELS with ≥15 fines	Silty gravels, poorly-graded gravel-sand-silt mixtures	GM	
				Clayey gravels, poorly-graded gravel-sand-clay mixture	GC	
		SANDS ≥50% coarse fraction passes #4 sieve	SANDS with little or no fines	Well-graded sands, gravelly sands, little or no fines	SW	
				Poorly-graded sands, gravelly sands, little or no fines	SP	
			SANDS with ≥15% fines	Silty sands, poorly-graded sand-gravel-silt mixtures	SM	
				Clayey sands, poorly-graded sand-gravel-clay mixtures	SC	
	FINE-GRAINED SOILS ≥50% passes #200 sieve	SILTS AND CLAYS liquid limit <50		Inorganic silts and very fine sands, silty or clayey fine sands, silts with slight plasticity	ML	
				Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays	CL	
				Organic silts and clays of low plasticity	OL	
		SILTS AND CLAYS liquid limit >50		Inorganic silts, micaceous or diatomaceous fine sand or silt	MH	
Inorganic clays of high plasticity, fat clays				CH		
Organic silts and clays of medium-to-high plasticity				OH		
HIGHLY ORGANIC SOILS				Peat, humus, swamp soils with high organic content	PT	

SOIL TYPE MODIFIERS

Sand/Gravel		Silt/Clay	
Term	% fines	Term	% fines
trace	<5	trace	<5
with	5-15	with	15-30
clayey/silty	>15	sandy/gravelly	>30

NOTE: Well-graded (wide range of grain sizes) = poorly sorted;
poorly-graded (predominantly one grain size) = well sorted

3	COLOR	Assign color using Munsell Soil Color Chart (1992) if possible
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4	CONSISTENCY (Sils and clays)			
Term	Blow/ft*			Field Test (when blow counts not available)
	1.4"ID	2.0"ID	2.5"ID	
very soft	0-2	0-2	0-2	Easily penetrated several inches by thumb; extrudes when squeezed
soft	2-4	2-4	2-4	Easily penetrated one inch by thumb; molded by light pressure
medium stiff	4-8	4-8	4-8	Penetrated over 1/2 inch by thumb with moderate effort; molded by strong pressure
stiff	8-15	9-17	9-18	Indented by 1/2 inch by thumb but penetrated only with great effort
very stiff	15-30	17-39	18-42	Readily indented by thumbnail
hard	30-60	39-78	42-85	Indented with difficulty by thumbnail
very hard	>60	>78	>85	Thumbnail will not indent soil

* = 140 pound hammer dropped 30 inches

DENSITY (Sands and gravels)			
Term	Blow/ft*		
	1.4"ID	2.0"ID	2.5"ID
very loose	0-4	0-5	0-7
loose	4-10	5-12	7-18
medium dense	10-29	12-37	18-51
dense	29-47	37-60	51-86
very dense	>47	>60	>86



5	MOISTURE CONTENT	
	Term	Field Test
	Dry	Absence of moisture, dusty, dry to the touch
	Moist	Damp but no visible water
	Wet	Visible free water

6	GRAIN SIZE		
	Term	Sieve size	Grain size
	Boulders	12 inches	>12 inches
	Cobbles	3-12 inches	3-12 inches
	Gravel - Coarse	3/4-3 inches	3/4-3 inches
	Fine	#4-3/4 inches	0.19-0.75 inches
	Sand - Coarse	#10-#4	0.079-0.19 inches
	Medium	#40-#10	0.017-0.079 inches
	Fine	#200-#40	0.0029-0.017 inches
	Fines	Passing #200	<0.0029 inches

7	CEMENTATION	
	Term	Field Test
	Weak	Crumbles or breaks with handling or slight finger pressure
	Moderate	Crumbles or breaks with considerable finger pressure
	Strong	Will not crumble or break with finger pressure

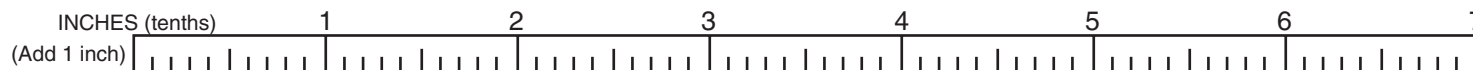
8	PLASTICITY	
	Nonplastic	Thread (1/8 inch or 3mm) cannot be rolled at any water content.
	Low	Thread can barely be rolled. Lump cannot be formed when drier than the plastic limit.
	Medium	Thread is easy to roll and not much time is required to reach the plastic limit. Thread cannot be rerolled after reaching the plastic limit. Lump crumbles when drier than the plastic limit.
	High	Takes considerable time rolling and kneading to reach the plastic limit. Thread can be rerolled several times after reaching the plastic limit. Lump can be formed without crumbling when drier than the plastic limit

9	MISCELLANEOUS		
	Plasticity (if applicable)	Fill or native material	Loss of drilling fluid
	Organics, carbon, vegetation, debris	Degree of rounding/angularity	Caving/sloughing
	Structure (e.g., layering)	Stratigraphic unit (if known)	Odor (organic, petroleum, or chemical)
	Coloration (staining, oxidation, mottling)	Drilling rate and rig behaviour	Organic vapor readings
	Lithology (e.g., quartz, mafic minerals)	Heaving sands	Fracturing

ROCK CLASSIFICATION						
Rock name	Color	Weathering	Fracturing	Competency	Mineralogy	Miscellaneous

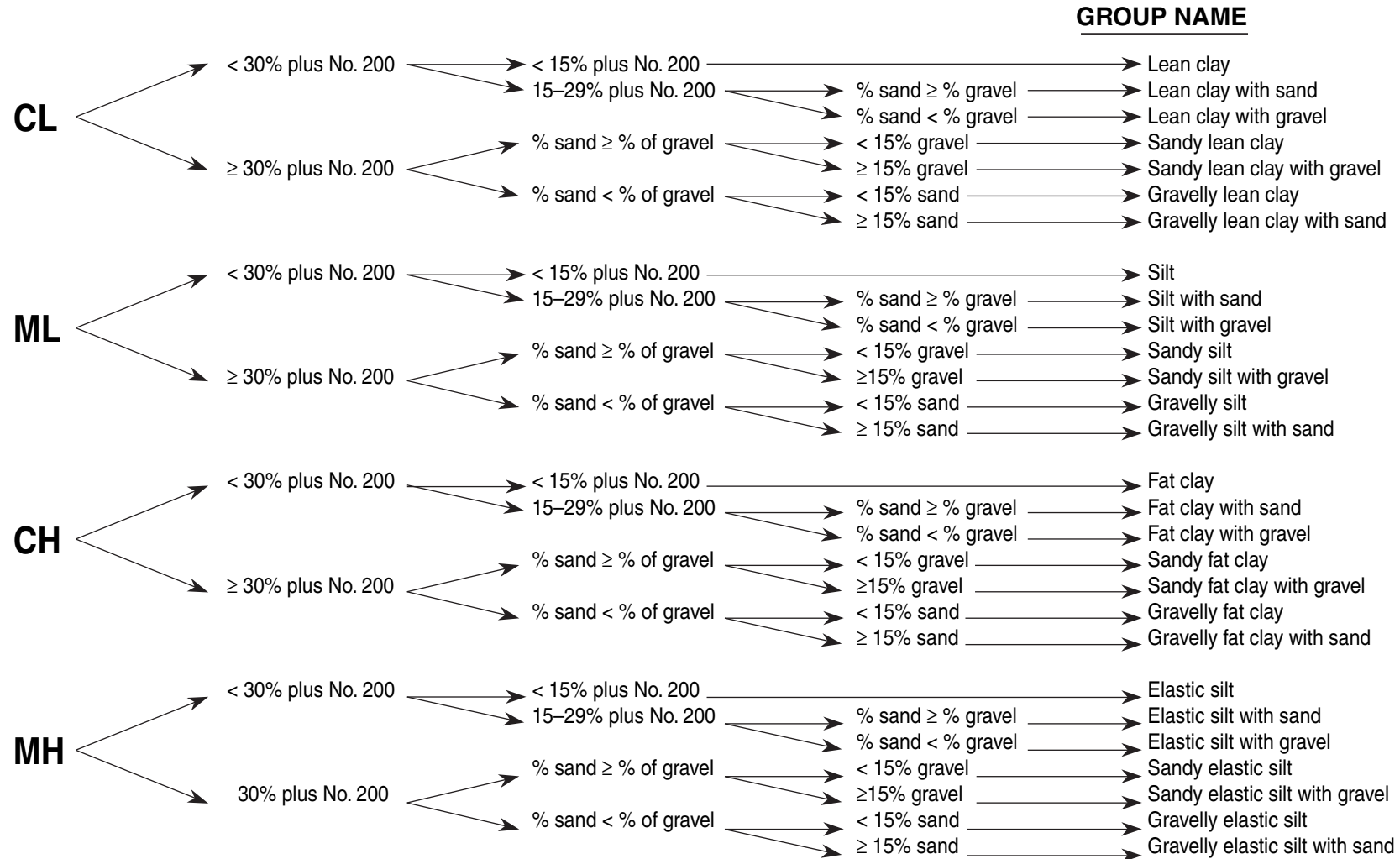
▽ Depth to first water (time and date)

▼ Depth to water after drilling (time and date)

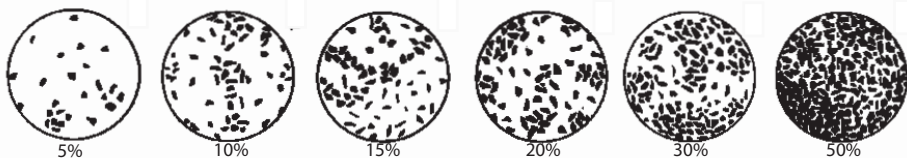


UNIFIED SOIL CLASSIFICATION SYSTEM FIELD GUIDE

Flow Chart for Identifying Fine-Grained Soils (more than 50% fines)



Estimated Percentages

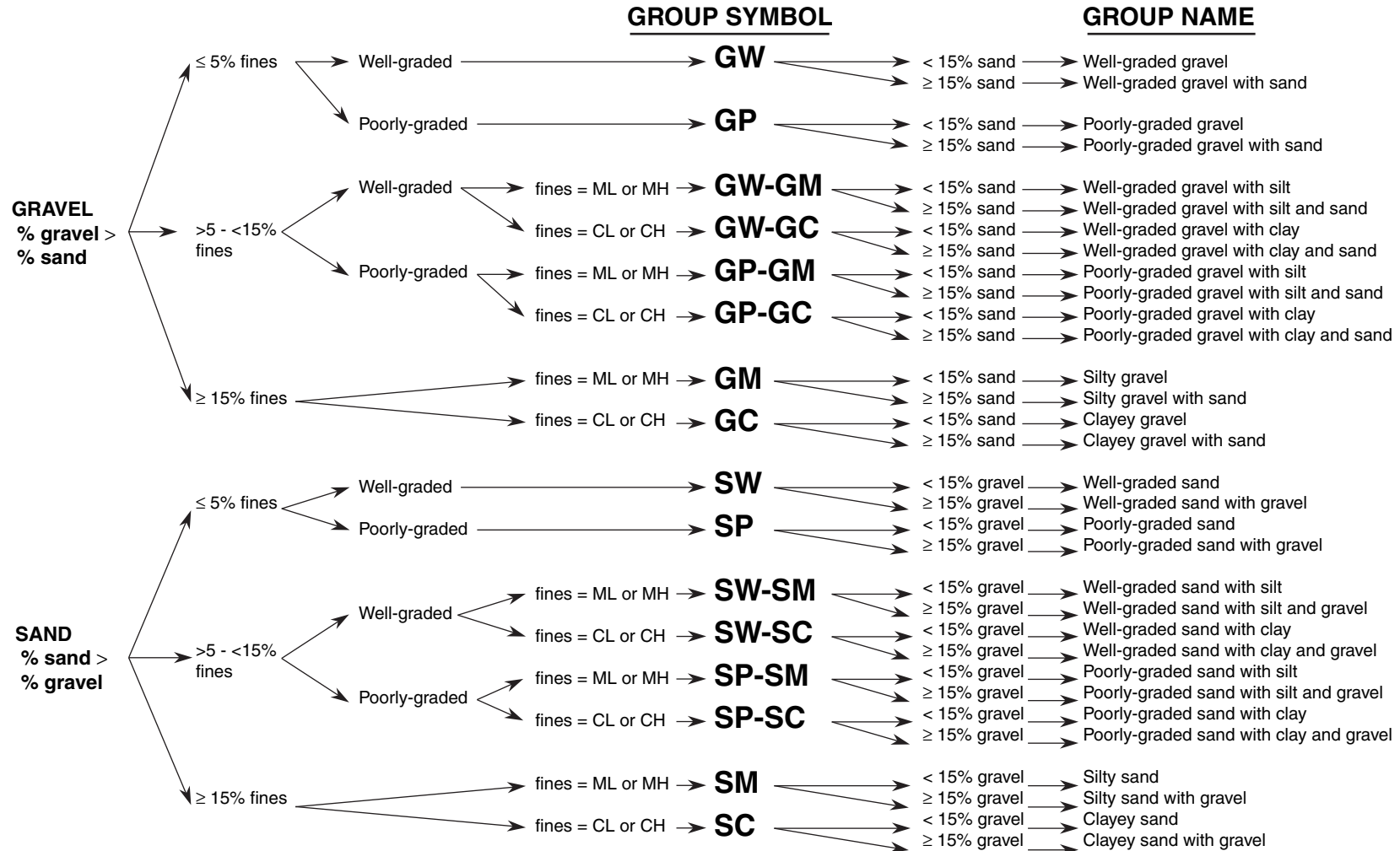


NOTES: — Percentages are based on estimating amounts of fines, sand and gravel to the nearest 5%.
— Material passing a No. 200 sieve is classified as fine; material retained on a No. 200 sieve is classified as sand and coarse-grained particles.

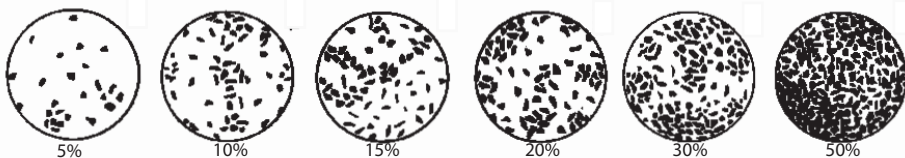


UNIFIED SOIL CLASSIFICATION SYSTEM FIELD GUIDE

Flow Chart for Identifying Coarse-Grained Soils (less than 50% fines)



Estimated Percentages



NOTES: — Percentages are based on estimating amounts of fines, sand and gravel to the nearest 5%.
— Material passing a No. 200 sieve is classified as fine; material retained on a No. 200 sieve is classified as sand and coarse-grained particles.



ATTACHMENT D
SOIL BORING LOG FORM

ATTACHMENT E
CRITERIA FOR DESCRIBING PLASTICITY

CRITERIA FOR DESCRIBING PLASTICITY	
Description	Criteria
Nonplastic	A 1/8-in. (3-mm) thread cannot be rolled at any water content
Low	The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit
Medium	The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit.
High	It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rerolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit.

ATTACHMENT F

CRITERIA FOR DESCRIBING DENSITY AND CONSISTENCY

DENSITY/CONSISTENCY BASED UPON BLOW COUNTS							
Density (Sand and Gravel) Blows/ft*				Consistency (Silt and Clay) Blows/ft*			
Term	1.4" ID	2.0" ID	2.5" ID	Term	1.4" ID	2.0" ID	2.5" ID
very loose	0-4	0-5	0-7	very soft	0-2	0-2	0-2
loose	4-10	5-12	7-18	soft	2-4	2-4	2-4
medium dense	10-29	12-37	18-51	medium stiff	4-8	4-9	4-9
dense	29-47	37-60	51-86	stiff	8-15	9-17	9-18
very dense	>47	>60	>86	very stiff	15-30	17-39	18-42
				hard	30-60	39-78	42-85
				very hard	>60	>78	>85
* 140 lb. hammer dropped 30 inches							

CRITERIA FOR DESCRIBING CONSISTENCY BASED UPON THUMB TEST	
Description	Criteria
Very soft	Thumb will penetrate soil more than 1 in. (25 mm)
Soft	Thumb will penetrate soil about 1 in. (25 mm)
Firm	Thumb will indent soil about 1/4 in. (6 mm)
Hard	Thumb will not indent soil but readily indented with thumbnail
Very Hard	Thumbnail will not indent soil

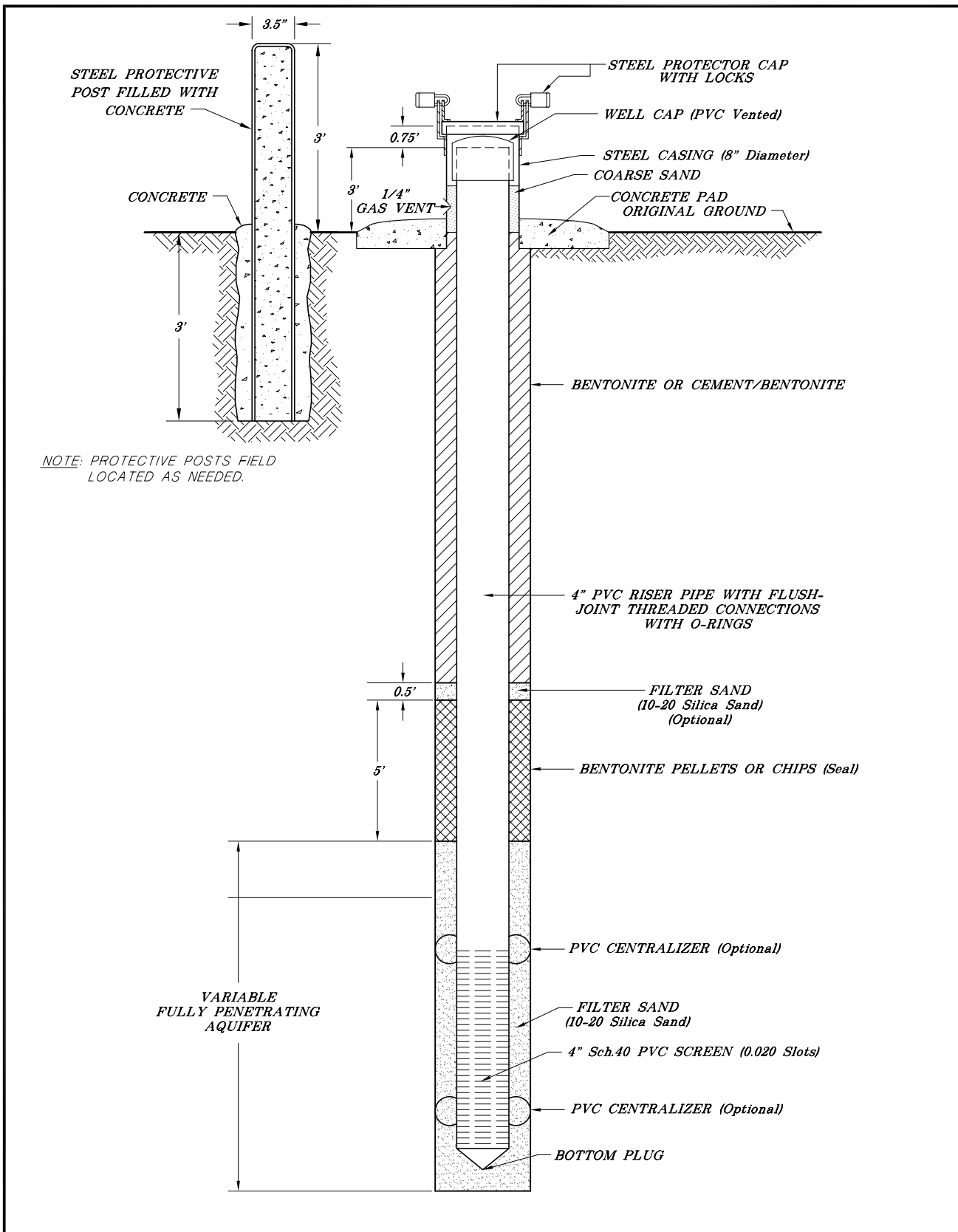
ATTACHMENT G


CRITERIA FOR DESCRIBING STRUCTURE

CRITERIA FOR DESCRIBING STRUCTURE	
Description	Criteria
Stratified	Alternating layers of varying material or color with layers at least 6 mm thick; note thickness
Laminated	Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness
Fissured	Breaks along definite planes of fracture with little resistance to fracturing
Slickensided	Fracture planes appear polished or glossy, sometimes striated
Blocky	Cohesive soil that can be broken down into small angular lumps which resist further breakdown
Lensed	Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness
Homogeneous	Same color and appearance throughout

ATTACHMENT H

TYPICAL MONITOR WELL INSTALLATION FORM



Project No.:	Design By:	Scale:	
		N.T.S.	
File:	Drawn By:	Date:	
TYP-WELL.DWG			
 MWH			TYPICAL MONITORING WELL INSTALLATION

ATTACHMENT I

MONITORING WELL CONSTRUCTION FORM

Project Name:		Date Drilled: From to		Well ID
Project Number:		Date Installed: From to		
Longitude:		Latitude:		Elevation:
Datum:				
Protective Casing:		BGS		AGS
Well Casing Top:		BGS		AGS
Well Casing Schedule: 40/80 Other:				
Well Casing Diameter:		in		
Screen Joint:		(BGS)		
Screen Material:		Manufacturer:		
Screen Type:		Slot Size:		
End Cap Bottom:		(BGS)		
Borehole Bottom:		(BGS)		
Borehole Diameter:		(BGS)		
Surface Seal (BGS): Top:		Bottom:		
Surface Seal Material:				
Annular Seal (BGS): Top:		Bottom:		
Annular Space Seal Material:				
Primary Filter Pack (BGS) Top:		Bottom:		
Sand Size:		Volume Added: Ft ³		
Manufacturer:		Cap and Lock:		
Protective Casing:				
Inside diameter:				
Drainage ports:				
Backfilled with:		From: to (BGS)		
Drilling Method:				
Drilling Fluids:				
Drilling Additives: (Describe)				
Water level after completion:		BGS		TOC
USCS Soil Classification @ Screen:				
GP GM GC GW SW SP		Bed Rock		
SM SC ML MH CL CH				
Bedrock Classification:				
Formation/Unit @ Screen:				
Well Drilled By			Well Installed By	
Firm:			Firm:	
Operator:			Installer:	
Notes:				
All lengths and depths are recorded in feet unless otherwise stated.				

ATTACHMENT J

WATER LEVEL READINGS FORM

[illegible]

Record of Water Level Readings, Rev: 4/10/2008



MWH

BUILDING A BETTER WORLD

SOP-2

SUBSURFACE SOIL SAMPLING PROCEDURES

SOP-2

SUBSURFACE SOIL SAMPLING PROCEDURES

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**STANDARD OPERATING PROCEDURES FOR
SUBSURFACE SOIL SAMPLING**

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1.0 INTRODUCTION

This standard operating procedure (SOP) is a general reference for the proper equipment and techniques for the collection of subsurface soil samples. The purpose of these procedures is to enable the user to collect representative and defensible subsurface soil samples for chemical analyses, and to facilitate planning of the field sampling effort. These techniques will be followed whenever applicable, although site-specific conditions or project-specific data quality objectives may require adjustments in methodology.

This SOP focuses on methods and equipment that are readily available and typically used for subsurface soil sample collection. It is not intended to provide an all-inclusive discussion of subsurface soil sampling methods.

2.0 DEFINITIONS

Brass Sleeve: Hollow, cylindrical, open-ended tubes used as liners in split-spoon samplers for the collection of undisturbed samples.

Continuous Core Barrel: 2.5 - 5 foot long steel barrels that can be joined together to allow continuous cores up to 60 feet long to be collected during a single run.

Hand Auger: A sampling tool consisting of a metal tube with two sharpened spiral wings at the tip.

Headspace: Free airspace in a sample container.

Split-Spoon Sampler: A sampling tool consisting of a thick-walled steel tube with a removable head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are removed.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and associated responsibilities. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

MWH Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Manager: Overall management responsibility for the sampling methods, sample locations, and constituents to be analyzed with input from other key project staff and Client personnel.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program and supervises other sampling personnel. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

4.0 SUBSURFACE SOIL SAMPLING PROCEDURES

The purpose of this SOP is to present methods for the collection of subsurface soil samples that will be used for environmental site characterization. Generally, subsurface soil samples are collected for chemical and/or geotechnical analysis.

4.1 Chemical Analysis

Chemical analysis of subsurface soil is conducted to assess the chemical properties of the subsurface materials. This information is used for site characterization and defining remedial design parameters. The samples are typically collected in relatively small quantities, often in several containers. Because the samples will be subjected to chemical analyses, prevention of cross-contamination during the sampling effort is critical.

4.2 Health and Safety Considerations

All sampling operations will be conducted in a manner that complies with the project-specific health and safety plan. Thus, during the collection of samples, health and safety information will be collected as required to ensure the safety of the sampling personnel.

Sampling personnel will wear personal protective equipment that is appropriate for the sampling location, media, and contaminants of concern, as determined in the site-specific health and safety plan. At a minimum, this will include clean, disposable, waterproof gloves to prevent cross contamination between samples or skin contact with possible contaminants. Additional safety equipment, including waterproof boots, coveralls, splash shields, respirators, etc., will be worn based on existing conditions and requirements of the project-specific health and safety plan.

4.3 Other Considerations

During sampling, care will be taken to ensure that the resulting data are representative of site conditions. If conditions exist prior to the collection of a sample which suggest that

materials from different stratigraphic units may have mixed (e.g., fill from a shallower depth that has sloughed into a hole and contacted soil at a greater depth), the hole or area will be thoroughly cleaned before sampling. It is critical that samples be representative of the materials scheduled to be sampled.

In addition, some individual sampling plans may require the collection of samples from a particular stratigraphic unit or layer rather than a particular depth. If, in these cases, a visual examination indicates that material from another layer has mixed with the sample, the non-desirable material will be separated from the sample or the sample will be discarded and re-collected.

4.4 Sampling Equipment and Methodology

Sampling equipment typically used to collect subsurface soil samples for chemical and geotechnical analyses with HAS drilling are listed below. Soil sample collection procedures using the listed types of equipment are outlined in the subsequent sections.

Split-Spoon Samplers: A split-spoon sampler consists of a thick steel tube with a ball check valve in a removable head and a removable hardened steel shoe. The barrel splits lengthwise to expose the sample when the head and shoe are removed. Split-spoon samplers are used to collect undisturbed samples for chemical, as well as geotechnical/engineering analyses.

Split-spoon samplers are typically 1.5 to 3 inches in outside diameter (OD), 18 or 24 inches in length and, if desired, may be lined on the interior with brass or acetate liners. Split-spoon samplers and brass liners will be decontaminated prior to use and stored in clean plastic bags until use. Decontamination procedures are described below. Both lined and unlined samplers are discussed below.

Split-Spoon Samplers with Liners: Brass or acetate liners may be placed inside the sampler in stacks. For sample collection, liners will be of a sufficient diameter to be retained within the sampler without obstructing the entry of a sample. The length of the sleeves will be specified in project-specific work plans.

To obtain a sample, the split-spoon will be lowered through the auger string or drill pipe to the underlying material and driven to the specified depth using a 140-pound (lb.) hammer falling 30 inches. The number of hammer blows required for every 6 inches of penetration will be recorded on the soil boring log during advancement of the sampler. Once the sample is obtained, the split-spoon will be removed from the hole and handled as appropriate to the type of sampling or compositing method required. For VOCs, the split-spoon will be opened in the field and samples collected according to the appropriate SOP. Samples for all other parameters may be submitted to the laboratory with the sample intact, or the sample can be extruded and transferred to appropriate sample containers. If a sample is submitted in the liner, the liner will then be capped on both ends.

Once the ends of the liner have been capped and sealed, the outside of the liner will be cleaned and the liner will be labeled appropriately. The label will be attached to the liner

in such a manner to ensure that the label is not lost. Self-adhesive tape may be used for this purpose. Once a sufficient number of liners have been collected for laboratory analyses, the remaining liners may be used for head-space analyses and sample logging, as necessary.

Split-Spoon Samplers without Liners: The procedure for the collection of soil samples from split-spoon samplers without liners is similar to that outlined for samplers with liners. The spoon is lowered through the auger string or drill pipe to the underlying material and driven to the specified depth using a 140-lb hammer falling 30 inches. The number of hammer blows required for every 6 inches of penetration will be recorded on the soil boring log during advancement of the sampler.

Once the sample is obtained, the split-spoon will be removed from the hole and handled according to the type of sampling or compositing method. For VOCs, the split-spoon will be opened in the field and samples collected according to appropriate SOP. Samples for all other parameters may be submitted to the laboratory with the sample intact, or the sample can be extruded and transferred to appropriate sample containers (as for PCBs and PAHs). Samples for VOC analysis will be collected as quickly as possible and placed in appropriate sample jars (refer to SOP for details). The sample will be collected in such a way as to minimize loss of volatile components.

5.0 DECONTAMINATION

All non-disposable equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Sample acquisition and compositing tools shall be decontaminated as follows:

1. Ensure that the cleaning solutions and rinseate containers required by governing sampling plans are available
2. Scrub the split-spoon sampler with a brush and rinse with deionized or distilled water.
3. Dispose of the rinseate and wiping rags in the manner specified in governing sampling plans.
4. Wrap the decontaminated device securely in clean plastic sheeting or bags pending next use.

Personnel shall don appropriate personal protective equipment as specified in the project-specific health and safety plan.

6.0 REFERENCES

U.S. Environmental Protection Agency (USEPA), 1986. Test Methods for Evaluating Solid Waste. SW-846 (Third Edition). Office of Solid Waste and Emergency Response. Washington, D.C.

SOP-3

SURFACE SOIL AND SUBSURFACE

SOIL SAMPLING PROCEDURES

FOR VOLATILE ORGANIC COMPOUND ANALYSIS

SOP-3

SURFACE SOIL AND SUBSURFACE SOIL SAMPLING PROCEDURES FOR VOLATILE ORGANIC COMPOUND ANALYSIS

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**STANDARD OPERATING PROCEDURES FOR
SURFACE AND SUBSURFACE SOIL SAMPLING
FOR VOLATILE ORGANIC COMPOUND ANALYSIS**

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1.0 INTRODUCTION

This standard operating procedure (SOP) describes methods and equipment that shall be used for collecting environmental surface soil, subsurface soil, and sediment samples for volatile organic compound (VOC) analysis. This SOP defines sample collection procedures for screening and definitive sampling levels, using a soil sampler, methanol, and sodium bisulfate preservation methods according to Method 5035A (attached to this SOP). This document focuses on methods and equipment that are specific to sampling surface soil and subsurface soil analysis. It is not intended to provide an all-inclusive discussion of soil sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the MWH Project Manager, Client Project Manager, and the Quality Manager.

2.0 DEFINITIONS

Environmental Sample: A solid sample collected for VOC analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

TerraCore™ Soil Sampler: A disposable, volumetric sampling device. The TerraCore™ sampler collects soil samples for transfer in to vials.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and associated responsibilities. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

MWH Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

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Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program and supervises other sampling personnel. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the

actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

4.0 SURFACE SOIL, SUBSURFACE SOIL, AND SEDIMENT SAMPLING PROCEDURES

4.1 Background

Surface soil samples are typically collected from the ground surface to 6 inches below ground surface. Samples collected from greater than 6 inches below ground surface are considered subsurface soil samples.

4.2 Sampling Program Objectives

The objective of surface soil and subsurface soil is to characterize the VOC analytes and possibly identify potential sources of contaminants. Sampling objectives are typically diverse and dependent on the nature of the project-specific data quality objectives. Details pertaining to sample locations, number of samples, and type of analyses required, shall be presented in the Work Plan and Field Sampling Plan (FSP).

4.3 Sampling Equipment and Techniques

Soil samples shall be collected and transferred to a wide-mouth 4-ounce jar, or a vial with a chemical preservative using a syringe or Terra Core sampler. For each discrete VOC sample, the sample will be collected starting at the desired sampling interval to within a few inches stratigraphically below the desired interval (e.g., three-six inches starting at 9 ft bgs interval) within the split-spoon. The samples will then transferred to a wide-mouth 4-ounce jar or a vial with a chemical preservative using a syringe or Terra CoreTM sampler.

Terra CoreTM Sampler: The Terra Core is a one time use transfer tool, designed to easily take samples from hard packed soils and transfer them to the appropriate containers for in-field chemical preservation. The Terra Core transfers soil samples as described in USEPA SW-846 Method 5035. The steps for use are as follows:

1. Have ready a tared 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 grams of soil.
2. Wipe all soil or debris from the outside of the Terra CoreTM sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.
3. Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the tared 40ml VOA

vial containing the appropriate preservative, and extrude the sample by pushing the plunger down. Quickly place the lid back on the tared 40ml VOA vial.

Note: When capping the 40ml VOA vial, be sure to remove any soil or debris from the threads of the vial.

The soil samples must be of small enough particle size to use the syringe or Terra Core sampler. Soils that are conducive to using the syringe or Terra Core sampler include soils that are classified as clays, silts and fine to medium grain sands and some coarse grain sands. If larger particle size materials need to be collected, a 5.0 gram sample will be collected with a spatula or scoop and carefully placed into the vial.

Methanol or Sodium Bisulfate Preservation: Methanol or Sodium Bisulfate preservation is used with the Terra Core™ sampler. Refer to SW-846 Method 5035A (U.S. EPA, 1996) for full details on sample preservation. A sodium bisulfate preservative solution is used for the collection of soil samples in which the suspected VOC concentration is in the range of 0.5 to 200 micrograms per kilogram ($\mu\text{g/kg}$). For soil samples in which the VOC concentration is suspected to be greater than 200 $\mu\text{g/kg}$, either a bulk sample may be collected (the laboratory will add a water miscible solvent) or the sample is collected in a vial that contains a water-miscible organic solvent (methanol). Soil or sediment samples are collected following the procedures described below.

1. For low VOC concentration samples (0.5 – 200 $\mu\text{g/kg}$): Collect the soil or sediment sample according to the procedures defined in the project specific Field Sampling Plan (FSP). Collect approximately a 5.0 gram sample (weighed in the field) and place it in a pre-weighed vial that already contains a stirring bar and a sodium bisulfate preservative solution and that has a septum-sealed screw cap. The sample vial with solution may be available from the laboratory. After sampling, the vial shall be immediately sealed and shipped (on ice) to the laboratory for analysis.

Soil samples that contain carbonate minerals may effervesce when in contact with the sodium bisulfate. If this occurs, the two options can be considered: the sample will be collected in a vial containing laboratory grade DI water or the addition of 5 mL of organic-free reagent water to each vial can be considered. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes. If this still occurs, the sample shall be collected in an un-preserved vial or other sampling container.

2. For high VOC concentration samples (greater than 200 $\mu\text{g/kg}$): Collect the soil sample according to the procedures defined in the project specific FSP, then follow one of the two options below:

Option 1: Collect a bulk sample in a vial or other suitable container without preservative. Seal the container and ship it (on ice) to the laboratory for analysis. The laboratory will take a sample from the container and add the appropriate amount of preservative prior to analysis.

Option 2: Collect approximately a 5 gram sample (weighed in the field) and place the sample in a pre-weighed vial with a septum-sealed screw-cap that contains 5 milliliters (mL) of water-miscible organic solvent, (methanol). The vial can either be prepared by the laboratory or prepared in the field at the time of sampling. Five (5) grams \pm 0.5g of sample shall be transferred to the vial immediately after sample collection and in a manner that minimizes loss of VOCs using the procedures described in the project specific FSP. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C.

3. If the FSP calls for replicate samples, collect at least two replicate samples, one for replicate analysis, the other for percent moisture determination. The replicate samples should be collected from the same location or within close proximity to the location from which the original sample was collected.
4. Because the soil vial cannot be opened without compromising the integrity of the sample, at least one additional vial of sample may be collected for dry weight determination. This additional replicate must not contain methanol, since an aliquot will be used for dry weight determination.
5. All samples for VOC analysis shall be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice.

Oily Waste Samples: If oily waste samples are known to be soluble in methanol then sample vials may be used as described above. However, if oily waste samples are not known to be or are not soluble in methanol then the sample should be collected in an un-preserved vial.

5.0 DECONTAMINATION

All non-disposable equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Sample acquisition and compositing tools shall be decontaminated as follows:

1. Ensure that the cleaning solutions and rinseate containers required by governing sampling plans are available
2. Scrub the sample acquisition or compositing tool with a brush and rinse with deionized or distilled water.
3. Dispose of the rinseate and wiping rags in the manner specified in governing sampling plans.
4. Wrap the decontaminated device securely in clean plastic sheeting or bags pending next use.

Personnel shall don appropriate personal protective equipment as specified in the project-specific health and safety plan. Note that when handling the vials that contain methanol, methanol resistant gloves shall be worn.

6.0 REFERENCES

U.S. Environmental Protection Agency. 1996. SW-846 Method 5035A Revision 0, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples.

METHOD 5035A

CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR
VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES

1.0 SCOPE AND APPLICATION

1.1 This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260. The following compounds are appropriate for this sample preparation technique:

Compound	CAS No. ^a	Response	Stability
Acetone	67-64-1	ht	hs
Acetonitrile	75-05-8	pp	nd
Acrolein (Propenal)	107-02-8	pp	ms
Acrylonitrile	107-13-1	pp	hs
Allyl alcohol	107-18-6	ht	nd
Allyl chloride	107-05-1	c	ms
t-Amyl ethyl ether (TAEE)	919-94-8	c / ht	nd
t-Amyl methyl ether (TAME)	994-05-8	c / ht	hs
Benzene	71-43-2	c	hs
Benzyl chloride	100-44-7	c	nd
Bis(2-chloroethyl)sulfide	505-60-2	pp	nd
Bromoacetone	598-31-2	pp	nd
Bromochloromethane	74-97-5	c	hs
Bromodichloromethane	75-27-4	c	ms
Bromoform	75-25-2	c	hs
Bromomethane	74-83-9	c	hvs
n-Butanol	71-36-3	ht	nd
2-Butanone (MEK)	78-93-3	pp	hvs
t-Butyl alcohol	75-65-0	ht	nd
Carbon disulfide	75-15-0	pp	hvs
Carbon tetrachloride	56-23-5	c	hvs
Chloral hydrate	302-17-0	pp	nd
Chlorobenzene	108-90-7	c	hvs
Chlorodibromomethane	124-48-1	c	nd
Chloroethane	75-00-3	c	ms

(continued)

Compound	CAS No. ^a	Response	Stability
2-Chloroethanol	107-07-3	pp	nd
2-Chloroethyl vinyl ether	110-75-8	c	ls
Chloroform	67-66-3	c	hs
Chloromethane	74-87-3	c	hvs
Chloroprene	126-99-8	c	nd
Crotonaldehyde	4170-30-3	pp	nd
1,2-Dibromo-3-chloropropane	96-12-8	pp	ms
1,2-Dibromoethane	106-93-4	c	hs
Dibromomethane	74-95-3	c	hs
1,2-Dichlorobenzene	95-50-1	c	hs
1,3-Dichlorobenzene	541-73-1	c	ms
1,4-Dichlorobenzene	106-46-7	c	ms
<i>cis</i> -1,4-Dichloro-2-butene	1476-11-5	c	nd
<i>trans</i> -1,4-Dichloro-2-butene	110-57-6	pp	ls
Dichlorodifluoromethane	75-71-8	c	hs
1,1-Dichloroethane	75-34-3	c	hs
1,2-Dichloroethane	107-06-2	c	hs
1,1-Dichloroethene	75-35-4	c	hvs
<i>cis</i> -1,2-Dichloroethene	156-59-4	c	hs
<i>trans</i> -1,2-Dichloroethene	156-60-5	c	ms
1,2-Dichloropropane	78-87-5	c	hs
1,3-Dichloro-2-propanol	96-23-1	pp	nd
<i>cis</i> -1,3-Dichloropropene	10061-01-5	c	ls
<i>trans</i> -1,3-Dichloropropene	10061-02-6	c	ls
1,2,3,4-Diepoxybutane	1464-53-5	c	nd
Diethyl ether	60-29-7	c	nd
Diisopropyl ether (DIPE)	108-20-3	c / ht	hs
1,4-Dioxane	123-91-1	pp	nd
Ethylbenzene	100-41-4	c	hvs
Ethylene oxide	75-21-8	pp	nd
Ethyl methacrylate	97-63-2	c	ms
Ethyl <i>tert</i> -butyl ether (ETBE)	637-92-3	c / ht	hs
Hexachlorobutadiene	87-68-3	c	ms
2-Hexanone	591-78-6	pp	hvs
Iodomethane	74-88-4	c	nd
Isobutyl alcohol	78-83-1	ht / pp	nd
Isopropylbenzene	98-82-8	c	ms
Malononitrile	109-77-3	pp	nd
Methacrylonitrile	126-98-7	pp	hs
Methylene chloride	75-09-2	c	hs
Methyl methacrylate	80-62-6	c	ms
4-Methyl-2-pentanone (MIBK)	108-10-1	pp	ms
Methyl <i>tert</i> -butyl ether (MTBE)	1634-04-4	c / ht	hs
Naphthalene	91-20-3	c	ms
Nitrobenzene	98-95-3	c	nd

(continued)

Compound	CAS No. ^a	Response	Stability
2-Nitropropane	79-46-9	c	nd
N-Nitroso-di- <i>n</i> -butylamine	924-16-3	pp	nd
Paraldehyde	123-63-7	pp	nd
2-Pentanone	107-87-9	pp	nd
2-Picoline	109-06-8	pp	nd
1-Propanol	71-23-8	ht / pp	nd
2-Propanol	67-63-0	ht / pp	nd
̂-Propiolactone	57-57-8	pp	nd
Propionitrile (ethyl cyanide)	107-12-0	ht	nd
<i>n</i> -Propylamine	107-10-8	c	nd
Styrene	100-42-5	c	hvs
1,1,1,2-Tetrachloroethane	630-20-6	c	hs
1,1,2,2-Tetrachloroethane	79-34-5	c	nd
Tetrachloroethene	127-18-4	c	ms
Toluene	108-88-3	c	hs
<i>o</i> -Toluidine	95-53-4	pp	nd
1,2,4-Trichlorobenzene	120-82-1	c	hs
1,1,1-Trichloroethane	71-55-6	c	ms
1,1,2-Trichloroethane	79-00-5	c	hs
Trichloroethene	79-01-6	c	ms
Trichlorofluoromethane	75-69-4	c	ls
1,2,3-Trichloropropane	96-18-4	c	ls
Vinyl acetate	108-05-4	c	ls
Vinyl chloride	75-01-4	c	hvs
<i>o</i> -Xylene	95-47-6	c	hvs
<i>m</i> -Xylene	108-38-3	c	hvs
<i>p</i> -Xylene	106-42-3	c	hvs

^a Chemical Abstract Service Registry Number

- c = Adequate response by this technique
ht = Method analyte only when purged at 80°C
pp = Poor purging efficiency resulting in high Estimated Quantitation Limits
nd = Not determined
hs = High stability in preserved water samples (> 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ms = Medium stability in preserved water samples (15 - 60 days). Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information
ls = Low stability in preserved water samples (< 14 days), analyses should be performed as soon as possible.
hvs = Highly variable stability in preserved water samples. Longer holding times may be appropriate, see Appendix A, Table A.1 footnote and ref. 47 for additional information.

1.2 The low soil method utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are minimized. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 0.5 to 200 µg/kg range.

1.3 Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

1.4 Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent. These samples are also purged using Method 5030.

1.5 This method can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are significantly higher because of poor purging efficiency. The purging efficiency can be improved for water soluble analytes, e.g. ketones and alcohols, when purging at an elevated temperature of 80°C as compared to 20° or 40°C.

1.6 This method, in conjunction with Method 8015 (GC/FID), may be used for the analysis of the aliphatic hydrocarbon fraction in the light ends of total petroleum hydrocarbons, e.g., gasoline. For the aromatic fraction (BTX), use this method and Method 8021 (GC/PID). A total determinative analysis of gasoline fractions may be obtained using Method 8021 in series with Method 8015.

1.7 As with any preparative method for volatiles, samples should be screened to avoid contamination of the purge-and-trap system by samples that contain very high concentrations of purgeable material above the calibration range of the low concentration method. In addition, because the sealed sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots should be collected to allow for screening and reanalysis.

1.8 The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 8.2.2).

1.9 Analysts should consult the disclaimer statement at the front of the manual and the information in Chapter Two for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.10 Use of this method is restricted to use by, or under supervision of, appropriately experienced and trained laboratory analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to soils and other solid samples with VOC concentrations in the range of 0.5 to 200 µg/kg (refer to Appendix A for additional information).

Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. To ensure minimal loss of volatile constituents prior to analysis the entire sample vial is placed, unopened with an unpierced septum, into the instrument auto sampler device. Immediately before analysis, organic-free reagent water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg (refer to Appendix A for additional information).

The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/ELCD, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect an appropriate sample volume in a pre-weighed vial with a septum-sealed screw-cap (see Sec 6) that contains a water-miscible organic solvent (e.g., methanol). At the time of analysis, an aliquot of the solvent is removed from the vial and diluted into water along with the internal standards and surrogates, then purged using Method 5030 and analyzed by an appropriate determinative method.

2.2.2 The second option is to collect a bulk sample in a VOA vial without the use of a chemical preservative. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent to dissolve the volatile organic constituents. An aliquot of the solution is added to reagent water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method. Because the procedure involves opening the vial and removing a portion of the soil, a significant amount of volatile constituents may be lost during handling. (See Appendix A, Sec. 5.1 for additional details)

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent.

Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol or polyethylene glycol (PEG), a separate aliquot of the sample is spiked with surrogates and diluted in the appropriate solvent. An aliquot of the solution is added to 5 mL of reagent water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) are added to the solution which is then purged using Method 5030 and analyzed by an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared according to Method 3585.

3.0 DEFINITIONS

Refer to Chapter One for a listing of applicable quality assurance/quality control (QA/QC) definitions.

4.0 INTERFERENCES

4.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which can be concentrated in the trap during the purge operation. These compounds can result in interferences or false positives in the determinative step.

4.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from an appropriate organic-free matrix and sample container, and carried through sampling and handling protocols, serves as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free reagent water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free reagent water is not necessary.

4.4 The laboratory where volatile analysis is performed should be completely free of solvents. Special precautions must be taken when analyzing for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed can also lead to random background levels and the same precautions must be taken.

5.0 SAFETY

This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals included in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

6.0 EQUIPMENT AND SUPPLIES

6.1 Sample containers

The specific sample containers required will depend on the purge-and-trap system to be employed (see Sec. 6.2). Several systems are commercially available. Some systems employ 40-mL clear vials with a special frit and equipped with two PTFE-faced silicone septa. Other systems permit the use of any good quality glass vial that is large enough to contain at least 5 g of soil or solid material and at least 10 mL of water and that can be sealed with a screw-cap containing a PTFE-faced silicone septum. Consult the purge-and-trap system manufacturer's instructions regarding the suitable specific vials, septa, caps, and mechanical agitation devices. Additional information on sample containers can be found in Appendix A, Secs. 1.6, 3.0, 7.0 and 8.0.

6.2 Purge-and-trap system

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards (if applicable) to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. Such systems are commercially available from several sources and shall meet the following specifications.

6.2.1 The purging device should be capable of accepting a vial sufficiently large enough to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device should also be capable of introducing at least 5 mL of organic-free reagent water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 6.2.2).

NOTE: The equipment used to develop this method was a Dynatech PTA-30 W/S Autosampler. This device was subsequently sold to Varian, and is now available as the Archon Purge and Trap Autosampler. See the Disclaimer at the front of this manual for guidance on the use of alternative equipment.

6.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed, it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all desired target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocabarb 4000, Supelco, Inc., Bellefonte, PA), as some degradation has been noted when higher desorption temperatures (especially above 240 - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocabarb 4000 but performs adequately when Vocabarb 3000 (Supelco, Inc., Bellefonte, PA) is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

6.2.2.1 The trap used to develop this method was 25 cm long, with an inside diameter of 0.105 inches, and was packed with Carboxpack/Carboxieve (Supelco, Inc., Bellefonte, PA).

6.2.2.2 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 25 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35°C are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

6.2.2.2.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

6.2.2.2.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

6.2.2.2.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

6.2.2.3 Trapping materials other than those listed above also may be employed, provided that they meet the specifications as noted above.

6.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

6.3 Syringe and syringe valves

6.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

6.3.2 2-way syringe valves with Luer ends.

6.3.3 25- μ L micro syringe with a 2-inch x 0.006-inch ID, 22° bevel needle (Hamilton #702N or equivalent).

6.3.4 Micro syringes - 10-, 100- μ L.

6.3.5 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight with shut-off valve.

6.4 Miscellaneous

6.4.1 Glass vials

6.4.1.1 60-mL, septum-sealed, to collect samples for screening, moisture determination.

6.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

6.4.2 Top-loading balance - Capable of accurately weighing to 0.01 g.

6.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

6.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground-glass stoppers.

6.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

6.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

6.4.7 Disposable Pasteur pipettes.

6.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

6.5 Field sampling equipment

6.5.1 Purge-and-trap soil sampler - Model 3780PT (Associated Design and Manufacturing Company, Alexandria, VA), or equivalent.

6.5.2 EnCore™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.3 Terra Core™ sampler - (En Novative Technologies, Inc., Green Bay, WI), or equivalent.

6.5.4 EasyDraw™ syringe and PowerStop™ handle - (US Oil Company, Inc., Kimberly, WI), or equivalent.

6.5.5 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

6.5.4 Portable balance - For field use, capable of weighing to 0.01 g.

6.5.5 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, reagent water added, cap, and septum.

6.5.6 Additional types of field sampling equipment and accessories are described in Appendix A, Secs. 1.6 and 7.0.

7.0 REAGENTS AND STANDARDS

7.1 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One.

7.2 Methanol, CH₃OH - purge-and-trap quality or equivalent. Store away from other solvents.

7.3 Polyethylene glycol (PEG), H(OCH₂CH₂)_nOH - free of interferences at the detection limit of the target analytes.

7.4 Low concentration sample preservative

7.4.1 For determination as to whether sample preservation is necessary and for selection of appropriate preservation options, see Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0.

7.4.2 Sodium bisulfate, NaHSO₄ - ACS reagent grade or equivalent.

7.4.3 The preservative, if necessary, should be added to the vial prior to shipment to the field, and must be present in the vial prior to adding the sample.

7.5 See the determinative method and Method 5000 for guidance on internal standards and surrogates to be employed in this procedure. The recommended surrogates are 4-bromofluorobenzene, 1,2-dichloroethane-d₄, and toluene-d₈. Other compounds may be used as surrogates, depending upon the analysis requirements and the specific target analytes. The recommended internal standards are chlorobenzene-d₅, 1,4-dichlorobenzene-d₄, and fluorobenzene. Other compounds may be used as internal standards as long as they have retention times similar to the target analytes being detected.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Refer to the introductory material in this chapter, Organic Analytes, Sec. 4.1, and Appendix A for general sample collection information. The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process.

As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

8.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.1 Low concentration soil samples

The following steps apply to the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in this method.

8.1.1.1 Add a clean magnetic stirring bar to each clean vial. If the purge-and-trap device (Sec. 6.2) employs a means of stirring the sample other than a magnetic stirrer (e.g., sonication or other mechanical means), then the stir bar is omitted.

8.1.1.2 Add preservative, if necessary, (See Appendix A, Secs. 1.2, 1.3, 3.0 and 8.0) to each vial. The preservative is added to each vial prior to shipping the vial to the field. Add approximately 1 g of sodium bisulfate to each vial. If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample. Enough sodium bisulfate should be present to ensure a sample pH of ≤ 2 .

8.1.1.3 Add 5 mL of organic-free reagent water to each vial. The water and the preservative will form an acid solution that will reduce or eliminate the majority of the biological activity in the sample, thereby preventing biodegradation of the volatile target analytes.

8.1.1.4 Seal the vial with the screw-cap and septum seal. If the double-ended, fritted, vials are used, seal both ends as recommended by the manufacturer.

8.1.1.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.1.6 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

8.1.1.7 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards (if applicable) should only be added to the vials after the sample has been added to the vial. These standards should be introduced back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

8.1.2 High concentration soil samples collected without a preservative

When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 60-mL glass vials with septum seals (see Sec. 6.4). More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3 High concentration soil samples collected and preserved in the field

The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030. See the water-miscible solvent dilution effect information in Sec. 11.5 and Method 8000 for guidance on correcting results for data reporting purposes. More detailed information on additional options for the preparation of sample vials can be found in Appendix A, Secs. 3.0, 7.0, and 8.0.

8.1.3.1 Add 10 mL of methanol to each vial.

8.1.3.2 Seal the vial with the screw-cap and septum seal.

8.1.3.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

8.1.3.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

8.1.3.5 Surrogates, internal standards and matrix spikes (if applicable) should be added to the sample after it is returned to the laboratory and prior to analysis.

8.1.4 Oily waste samples

When oily waste samples are known to be soluble in methanol or PEG, sample vials may be prepared as described in Sec. 8.1.3, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 8.1.2.

8.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™, or any other sampling device listed in Sec. 6.5, or equivalent. Always wear gloves whenever handling the tared sample vials. More detailed information and additional sample collection options can be found in Appendix A, Sec. 7.0.

8.2.1 Low concentration soil samples

8.2.1.1 Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample and shipping to the laboratory or appropriate analysis site by the various methods outlined in Appendix A. Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.1.2 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the sample vial containing the preservative solution or other preservation options as discussed in Appendix A. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4°C. Alternatively, samples can be collected into an empty vial or vial containing reagent water (with or without preservative) and stored frozen at < -7°C.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution option in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials without chemical preservation.

8.2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.1.4 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.1.5 As with the collection of aqueous samples for volatiles, collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis, if needed. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.

8.2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, moisture determination, and high concentration analysis (if necessary). This third aliquot may be collected in a 60-mL glass vial or a third 40-mL soil sample vial. However, this third vial must *not* contain the sample preservative solution, as an aliquot will be used to determine % moisture. If high concentration samples are collected in

vials containing methanol, then two additional aliquots should be collected, one for high concentration analysis collected in a vial containing methanol, and another for the moisture determination in a vial without either methanol or the low concentration aqueous preservative solution.

8.2.1.7 If samples are known or expected to contain target analytes over a wide range of concentrations, thereby requiring the analyses of multiple sample aliquots, it may be advisable and practical to take an additional sample aliquot in a low concentration soil vial containing the preservative, but collecting only 1-2 g instead of the 5 g collected in Sec. 8.2.1.1. This aliquot may be used for those analytes that exceed the instrument calibration range in the 5-g analysis.

8.2.1.8 The EnCore™ sampler has not been thoroughly evaluated by EPA as a sample storage device. While preliminary results indicate that storage in the EnCore™ device may be appropriate for up to 48 hours, samples collected in this device should be transferred to the soil sample vials as soon as possible, or analyzed within 48 hours.

8.2.1.9 The collection of low concentration soil samples in vials that contain methanol is not appropriate for samples analyzed with the closed-system purge-and-trap equipment described in this method (see Sec. 8.2.2).

8.2.2 High concentration soil samples preserved in the field

The collection of soil samples in vials that contain methanol has been suggested by some as a combined preservation and extraction procedure. However, this procedure is not appropriate for use with the low concentration soil procedure described in this method.

NOTE: The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of three potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 µg/kg). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 100, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. Secondly, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (see Sec. 11.5 and Method 8000) The final problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, or cause it to become a listed waste, thereby requiring the unused sample volume to be managed as a hazardous waste.

8.2.2.1 When samples are known to contain volatiles at concentrations high enough that the dilution factor will not preclude obtaining results within the calibration range of the appropriate determinative method, a sample may be collected and immediately placed in a sample vial containing purge-and-trap grade methanol.

8.2.2.2 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

8.2.2.3 Using the sample collection device, add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap. Store samples on ice at 4 °C.

8.2.2.4 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed (Sec. 6.5.5). Record the weight of the sealed vial containing the sample to the nearest 0.01 g.

8.2.2.5 Alternatively, collect several trial samples with plastic syringes. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length of soil in the syringe that corresponds to 5.0 ± 0.5 g. Discard each trial sample.

8.2.2.6 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.

8.2.2.7 The collection of at least one additional sample aliquot is required for the determination of the moisture content, as described in Sec. 6.2.1.6. Samples collected in methanol should be shipped as described in Sec. 6.3, and must be clearly labeled as containing methanol, so that the samples are not analyzed using the closed-system purge-and-trap equipment described in this procedure.

8.2.3 High concentration sample not preserved in the field

The collection of high concentration bulk samples, i.e., wastes containing percent level concentrations, that are not preserved in the field generally follows similar procedures as for the other types of samples described in Secs. 8.2.1 and 8.2.2, with the obvious exception that the sample vials contain neither the aqueous preservative solution nor methanol. However, when field preservation is not employed, it is better to collect a larger volume sample, filling the sample container as full as practical in order to minimize the headspace. Such collection procedures generally do not require the collection of a separate aliquot for moisture determination, but it may be advisable to collect a second sample aliquot for screening purposes, in order to minimize the loss of volatiles in either aliquot.

8.2.4 Oily waste samples

The collection procedures for oily samples depend on knowledge of the waste and its solubility in methanol or other solvents.

8.2.4.1 When an oily waste is known to be soluble in methanol or PEG, the sample may be collected in a vial containing such a solvent (see Sec. 8.1.4), using procedures similar to those described in Sec. 8.2.2.

8.2.4.2 When the solubility of the oily waste is not known, the sample should either be collected in a vial without a preservative, as described in Sec. 8.2.3, or the

solubility of a trial sample should be tested in the field, using a vial containing solvent. If the trial sample is soluble in the solvent, then collect the oily waste sample as described in Sec. 8.2.2. Otherwise, collect an unpreserved sample as described in Sec. 8.2.3.

8.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample handling options.

8.4 Sample storage

8.4.1 Once in the laboratory, store samples at the recommended temperature until analysis (refer to Appendix A, Secs. 3.0 and 7.4 for additional sample storage information). The sample storage area should be free of organic solvent vapors.

8.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

8.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, storage of low concentration samples at <-7°C (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples. Whichever steps are employed, they should be clearly described in the sampling and QA project plans and distributed to both the field and laboratory personnel. See Sec. 8.2.1.2 for additional information.

8.4.4 See Appendix A, Secs. 3.0, 7.0, and 8.0 for additional sample storage options.

9.0 QUALITY CONTROL

9.1 Refer to Chapter One for guidance on quality assurance (QA) and quality control (QC) protocols and Method 5000 for sample preparation QC procedures. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data generated. All data sheets and quality control data should be maintained for reference or inspection. When inconsistencies exist between QC guidelines, method-specific QC criteria take precedence over both technique-specific criteria and those criteria given in Chapter One, and technique-specific QC criteria take precedence over the criteria in Chapter One.

9.2 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free reagent water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

9.3 Initial demonstration of proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made. See the Quality Control Section of Methods 5000 and 8000 for information on how to accomplish this demonstration.

9.4 Sample quality control for preparation and analysis - See the Quality Control Section of Method 5000 and Method 8000 for procedures to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.5 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.6 The laboratory should have quality control procedures to make sure that sample integrity is not compromised during the sample collection and sample handling process, e.g., making sure that septa and vial caps do not leak, etc. (See Appendix A, Secs. 1.6 and 7.1.1) In addition, it would be advisable for the laboratory to monitor the internal standard's (IS) area counts for the low concentration samples, since leaks attributed to a poor seal with the vial caps and septa will be evident by low IS area counts. Sample containers and data results for instances where low IS area counts are observed and leaks are suspected, should be discarded.

10.0 CALIBRATION AND STANDARDIZATION

Refer to the appropriate determinative method for calibration and standardization procedures.

11.0 PROCEDURE

This section describes procedures for sample screening, the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

11.1 Sample screening

11.1.1 It is highly recommended that all samples be screened prior to the purge-and-trap GC or GC/MS analysis. Samples may contain higher than expected quantities of purgeable organics that will contaminate the purge-and-trap system, thereby requiring extensive cleanup and instrument maintenance. The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low concentration closed-system direct purge-and-trap method (Sec. 11.2), the high concentration (methanol extraction) method (Sec. 11.3), or the nonaqueous liquid (oily waste) methanol or PEG dilution procedure (Sec. 11.4).

11.1.2 The analyst may employ any appropriate screening technique. Three suggested screening techniques employing SW-846 methods are:

11.1.2.1 Automated headspace (Method 5021) using a gas chromatograph (GC) equipped with an appropriate detector,

11.1.2.2 Screening with a portable photoionization detector (PID) (Method 3815)
or,

11.1.2.3 Extraction of the sample with hexadecane (Method 3820) and analysis of the extract on a GC equipped with a FID and/or an ECD.

11.1.3 The analyst may inject a calibration standard containing the analytes of interest at a concentration equivalent to the upper limit of the calibration range of the low concentration soil method. The results from this standard may be used to determine when the screening results approach the upper limit of the low concentration soil method. There are no linearity or other performance criteria associated with the injection of such a standard, and other approaches may be employed to estimate sample concentrations.

11.1.4 Use the low concentration closed-system purge-and-trap method (Sec. 11.2) if the estimated concentration from the screening procedure falls within the calibration range of the selected determinative method. If the concentration exceeds the calibration range of the low concentration soil method, then use either the high concentration soil method (Sec. 11.3), or the oily waste method (Sec. 11.4).

11.2 Low concentration soil method (Approximate concentration range of 0.5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

11.2.1 Initial set-up

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated. General calibration procedures are discussed in Method 8000, while the determinative methods and Method 5000 provide specific information on calibration and preparation of standards. Normally, external standard calibration is preferred for the GC methods (non-MS detection) because of possible interference problems with internal standards. If interferences are not a problem, or when a GC/MS method is used, internal standard calibration may be employed.

11.2.1.1 Assemble a purge-and-trap device that meets the specification in Sec. 6.2 and that is connected to a gas chromatograph or a gas chromatograph/mass spectrometer system.

11.2.1.2 Before initial use, a Carboxpack/Carboxieve trap should be conditioned overnight at 245°C by baking out with an inert gas flow of at least 20 mL/minute. If other trapping materials are substituted for the Carboxpack/Carboxieve, follow the manufacturers recommendations for conditioning. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be conditioned by baking for 10 minutes at 245°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.3 If the standard trap in Sec. 6.2.2.2 is employed, prior to initial use, the trap should be conditioned overnight at 180°C by baking out with an inert gas flow of at least 20 mL/min, or according to the manufacturer's recommendations. Vent the trap effluent to the hood, not to the analytical column. Prior to daily use, the trap should be

conditioned by baking for 10 min at 180°C. The trap may be vented to the analytical column during daily conditioning; however, the column must be run through the temperature program prior to analysis of samples.

11.2.1.4 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 5 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

11.2.1.5 Prepare a minimum of five initial calibration standards containing all the analytes of interest and surrogates, as described in Method 8000, and following the instrument manufacturer's instructions. The calibration standards are prepared in organic-free reagent water. The volume of organic-free reagent water used for calibration must be the same volume used for sample analysis (normally 5 mL added to the vial before shipping it to the field plus the organic-free reagent water added by the instrument). When the sodium bisulfate preservation technique is used, the calibration standards should also contain approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~1 g), as the presence of the preservative will affect the purging efficiencies of the analytes. The internal standard solution must be added automatically, by the instrument, in the same fashion as used for the samples. Place the soil vial containing the solution in the instrument carousel. In order to calibrate the surrogates using standards at five concentrations, it may be necessary to disable the automatic addition of surrogates to each vial containing a calibration standard (consult the manufacturer's instructions). Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as recommended by the manufacturer.

11.2.1.6 Carry out the purge-and-trap procedure as outlined in Secs. 11.2.3. to 11.2.5.

11.2.1.7 Calculate calibration factors (CF) or response factors (RF) for each analyte of interest using the procedures described in Method 8000. Calculate the average CF (external standards) or RF (internal standards) for each compound, as described in Method 8000. Evaluate the linearity of the calibration data, or choose another calibration model, as described in Method 8000 and the specific determinative method.

11.2.1.8 For GC/MS analysis, a system performance check must be made before this calibration curve is used (see Method 8260). If the purge-and-trap procedure is used with Method 8021, evaluate the response for the following four compounds: chloromethane; 1,1-dichloroethane; bromoform; and 1,1,2,2-tetrachloroethane. They are used to check for proper purge flow and to check for degradation caused by contaminated lines or active sites in the system.

11.2.1.8.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

11.2.1.8.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response.

11.2.1.8.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

11.2.1.9 When analyzing for very late eluting compounds with Method 8021 (i.e., hexachlorobutadiene, 1,2,3-trichlorobenzene, etc.), cross-contamination and memory effects from a high concentration sample or even the standard are a common problem. Extra rinsing of the purge vessel after analysis normally corrects this. The newer purge-and-trap systems often overcome this problem with better bake-out of the system following the purge-and-trap process. Also, the charcoal traps retain less moisture and decrease the problem.

11.2.2 Calibration verification (see appropriate determinative method)

Refer to Method 8000 for details on calibration verification. A single standard near the mid-point of calibration range is used for verification. This standard should also contain approximately 1 g of sodium bisulfate if the samples are also preserved in this manner.

11.2.3 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. Consult the instrument manufacturer's instructions regarding larger sample sizes, in order to avoid clogging of the purging apparatus. The soil vial is hermetically sealed at the sampling site, and MUST remain so in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the exterior of the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

11.2.3.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

11.2.3.2 Without disturbing the hermetic seal on the sample vial, add 5 mL of organic-free reagent water, the internal standards, and the surrogate compounds. This is carried out using the automated sampler. Other volumes of organic-free reagent water may be used, however, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free reagent water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

11.2.3.3 For the sample selected for matrix spiking, add the matrix spiking solution described in the Reagents Section of Method 5000, either manually, or automatically, following the manufacturer's instructions. The concentration of the spiking solution and the amount added should be established as described in the Quality Control Section of Method 8000.

11.2.3.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for the appropriate purge time (usually 11 minutes) while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a glass-lined transfer line to a trap packed with suitable sorbent materials.

11.2.4 Sample desorption

11.2.4.1 Non-cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes (1.5 min is normally adequate for analytes in Method 8015). Begin the temperature program of the gas chromatograph and start data acquisition.

11.2.4.2 Cryogenic interface - After the purge, place the purge-and-trap system in the desorb mode, make sure that the cryogenic interface is at -150°C or lower, and rapidly heat the trap to 245°C while backflushing with an inert gas at 4 mL/minute for about 5 minutes (1.5 min is normally adequate for analytes in Method 8015). At the end of the 5-minute desorption cycle, rapidly heat the cryogenic trap to 250°C. Begin the temperature program of the gas chromatograph and start the data acquisition.

11.2.5 Trap reconditioning

After desorbing the sample, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

11.2.6 Data interpretation

Perform qualitative and quantitative analysis following the guidance given in the determinative method and Method 8000. If the concentration of any target analyte exceeds the calibration range of the instrument, it will be necessary to reanalyze the sample by the high concentration method. Such reanalyses need only address those analytes for which the concentration exceeded the calibration range of the low concentration method. Alternatively, if a sample aliquot of 1-2 g was also collected (see Sec. 8.2.1.7), it may be practical to analyze that aliquot for the analytes that exceeded the instrument calibration range in the 5-g analysis. If results are to be corrected for moisture content, proceed to Sec. 11.5.

11.3 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free reagent water containing, if applicable, internal and matrix spiking standards, purged according to Method 5030, and analyzed by an appropriate determinative method. Wastes that are insoluble in methanol (i.e., petroleum and coke wastes) are diluted with hexadecane (see Sec. 11.3.8).

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below,

beginning at Sec. 11.3.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 11.3.4.

11.3.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Whenever practical, mix the contents of the sample container by shaking or other mechanical means without opening the vial. When shaking is not practical, quickly mix the contents of the vial with a narrow metal spatula and immediately reseal the vial.

11.3.2 If the sample is from an unknown source, perform a solubility test preferably using a sample container reserved for the % moisture determination before proceeding. Remove several grams of material from the sample container. If the sample material is obtained from a vial dedicated for analysis, quickly reseal the container to minimize the loss of volatiles. Weigh 1-g aliquots of the sample into several test tubes or other suitable containers. Add 10 mL of methanol to the first tube, 10 mL of PEG to the second, and 10 mL of hexadecane to the third. Swirl the sample and determine if it is soluble in the solvent. Once the solubility has been evaluated, discard these test solutions. If the sample is soluble in either methanol or PEG, proceed with Sec. 11.3.3. If the sample is only soluble in hexadecane, proceed with Sec. 11.3.8.

11.3.3 For soil and solid waste samples that are soluble in methanol, add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution, or 10.0 mL of methanol without surrogates to a tared 20-mL vial. Using a top-loading balance, weigh 5 g (wet weight) of sample into the vial. Quickly cap the vial and reweigh the vial. Record the weight to 0.1 g. See Appendix A, Sec. 6.2.1 for methanol contact time information. If the sample was not soluble in methanol, but was soluble in PEG, employ the same procedure described above, but use 9.0 or 10.0 mL of PEG in place of the methanol. Proceed with Sec. 11.3.5.

NOTE: The steps in Secs. 11.3.1, 11.3.2, and 11.3.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

11.3.4 For soil and solid waste samples that were collected in methanol or PEG (see Sec. 8.2.2), weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum, and proceed with Sec. 11.3.5. See Appendix A, Sec. A.6.2.1 for methanol contact time information.

11.3.5 Pipet approximately 1 mL of the extract from either Sec. 11.3.3 or 11.3.4 into a GC vial for storage, using a disposable pipet, and seal the vial. The remainder of the extract may be discarded. Add approximately 1 mL of methanol or PEG to a separate GC vial for use as the method blank for each set of samples extracted with the same solvent.

11.3.6 The extracts must be stored at 4°C in the dark, prior to analysis. Add an appropriate aliquot of the extract (based on the approximate sample concentration as noted in the table below) to 5.0 mL of organic-free reagent water containing if applicable, surrogates, internal standards, and matrix spike compounds, and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to the Procedure Section in Method 5030 and follow the procedure for purging high concentration samples.

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF
HIGH CONCENTRATION SOILS/SEDIMENTS

Approximate Concentration Range			Volume of Methanol Extract ^a
500	-	10,000 µg/kg	100 µL
1,000	-	20,000 µg/kg	50 µL
5,000	-	100,000 µg/kg	10 µL
25,000	-	500,000 µg/kg	100 µL of 1/50 dilution ^b

Calculate appropriate dilution factor for concentrations exceeding those in this table.

- ^a The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5-mL syringe whatever volume of methanol is necessary to maintain a total volume of 100 µL of methanol.
- ^b Dilute an aliquot of the methanol extract and then take 100 µL for analysis.

11.3.7 If results are to be reported using a correction factor for moisture content, determine the moisture content of a separate aliquot of the sample, using the procedure in Sec. 11.5, after the sample extract has been transferred to a GC vial and the vial sealed.

11.3.8 For solids that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste) dilute or extract the sample with hexadecane using the procedures in the Procedure Section of Method 3585.

11.4 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free reagent water, purged according to Method 5030, and analyzed using an appropriate determinative method.

NOTE: Surrogate compounds may either be spiked into the solvent at the time of extraction or the reagent water containing an aliquot of the extract prior to analysis. Since the surrogate recovery data from these two options provides assurances of either extraction or analytical efficiencies, the decision as to when the surrogates are added depends on what questions need to be answered for a given sample matrix and the intended uses of the data.

For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane using the procedures in the Procedure Section of Method 3585.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 11.4.1. If methanol preservation was employed in the field, then the preparation begins with Sec. 11.4.3.

11.4.1 If the waste was not preserved in the field and it is soluble in methanol or PEG, weigh 1 g (wet weight) of the sample into a tared 10-mL volumetric flask, a tared scintillation vial, or a tared culture tube. If a vial or tube is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis.

11.4.1.1 To calibrate the vessel, pipet 10.0 mL of methanol or PEG into the vial or tube and mark the bottom of the meniscus.

11.4.1.2 Discard this solvent, and proceed with weighing out the 1-g sample aliquot.

11.4.2 Quickly add 1.0 mL of surrogate spiking solution, if desired, to the flask, vial, or tube, and dilute to 10.0 mL with the appropriate solvent (methanol or PEG). Swirl the vial to mix the contents. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.3 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field. If desired, add the surrogate spiking solution to the vial by injecting it through the septum. Swirl the vial to mix the contents and proceed with Sec. 11.4.4. See Appendix A, Sec. 6.2.1 for methanol contact time information.

11.4.4 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

11.4.5 Add 10 - 50 μ L of the methanol extract to 5 mL of organic-free reagent water containing if applicable, surrogates and internal standards, followed by purge-and-trap analysis, using Method 5030.

11.4.6 If necessary, prepare a matrix spike sample by adding 10 - 50 μ L of the matrix spike standard dissolved in methanol to a 1-g aliquot of the oily waste. Shake the vial to disperse the matrix spike solution throughout the oil. Then add 10 mL of extraction solvent and proceed with the extraction and analysis, as described in Secs. 11.4.2 - 11.4.5. Calculate the recovery of the spiked analytes as described in Method 8000. If the recovery is not within the acceptance limits for the application, use the hexadecane dilution technique in the Procedure Section of Method 3585.

11.5 Determination of % moisture

If results are to be reported using a correction factor for moisture content, it is necessary to determine the moisture content of the sample. Also note that solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) In order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

NOTE: It is highly recommended that the moisture content determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the moisture content determination. Thus, this determination can be made any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

11.5.1 Weigh 5-10 g of the sample from the 60-mL VOA vial into a tared crucible.

11.5.2 Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing. Calculate the % moisture as follows:

$$\% \text{ moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

WARNING: The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from a heavily contaminated hazardous waste sample.

12.0 DATA ANALYSIS AND CALCULATIONS

There are no calculations explicitly associated with this extraction procedure. See the appropriate determinative method and Method 8000 for calculation of final sample results.

13.0 METHOD PERFORMANCE

13.1 Single laboratory accuracy and precision data were obtained for the method analytes in three soil matrices, sand, a soil collected 10 feet below the surface of a hazardous landfill, called the C-Horizon, and a surface garden soil. Each sample was fortified with the analytes at a concentration of 20 ng/5 g, which is equivalent to 4 µg/kg. These data are listed in tables found in Method 8260.

13.2 Single laboratory accuracy and precision data were obtained for certain method analytes when extracting oily liquid using methanol as the extraction solvent. The data are presented in a table in Method 8260. The compounds were spiked into three portions of an oily liquid (taken from a waste site) following the procedure for matrix spiking described in Sec. 7.4. This represents a worst case set of data based on recovery data from many sources of oily liquid.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste*

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 12.2.

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APPENDIX A

THE COLLECTION AND PRESERVATION OF AQUEOUS AND SOLID SAMPLES FOR VOLATILE ORGANIC COMPOUND (VOC) ANALYSIS

FOREWORD

The information provided in this Appendix is based on EPA's evaluation of currently available data and technology as applied to the most appropriate sample handling and preservation procedures in order to minimize the loss of volatile organic compounds (VOCs) during the collection and analysis of aqueous and solid materials, such as groundwater, wastewater, soils, solid waste, or sediments. These procedures are designed to minimize the losses of VOCs through the two most common mechanisms, volatilization and biodegradation. The intended users of this Appendix guidance are those individuals and organizations involved in the collection and preparation of samples for VOC analyses during the characterization of solid materials under the Resource Conservation and Recovery Act (RCRA). The procedures and techniques described in this Appendix are not presented in any preferential order nor do they represent EPA requirements, but rather they are intended solely as guidance and should be selected and utilized based on the stated project-specific data quality objectives.

This Method 5035 Appendix was developed under the direction of Mr. Barry Lesnik, U.S. EPA, Office of Solid Waste (OSW), Methods Team in collaboration with Mr. David Payne, U.S. EPA, Region 5, Mr. Alan Hewitt, U.S. ACE CRREL, and the SW-846 Organic Methods Workgroup Members. The Methods Team is the focal point within OSW for expertise in analytical chemistry and characteristic testing methodologies, environmental sampling and monitoring, and quality assurance. The Methods Team provides technical support to other OSW Divisions, EPA Program Offices and Regions, state regulatory agencies, and the regulated community.

DISCLAIMER

The U.S. Environmental Protection Agency's Office of Solid Waste (EPA or the Agency) has prepared this Method 5035 Appendix to provide guidance to those individuals involved in the collection and preparation of samples for volatile organic compounds (VOCs) analysis during the characterization of aqueous and solid materials under the Resource Conservation and Recovery Act (RCRA). This Appendix provides guidance for selecting an appropriate sample collection and preservation technique that may be suitable for VOC analyses in order to meet the data quality requirements or objectives for the intended use of the results.

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A.1.0 PURPOSE AND OVERVIEW

This Appendix provides guidance in sample collection and preservation procedures that may be suitable for use during the characterization of volatile organic compounds (VOCs) in solid materials, such as soils, solid wastes, or sediments and aqueous samples or leachates from solid matrices.

A.1.1 What are VOCs?

VOCs are a class of organic compounds that includes low molecular weight aromatics, hydrocarbons, halogenated hydrocarbons, ketones, acetates, nitriles, acrylates, ethers, and sulfides with sufficiently low boiling points to give them appreciable vapor pressures at 1 atmosphere of pressure. Although EPA has never defined a strict boiling point cut-off for this compound class, most VOCs of concern to EPA have boiling points below 150 °C, while some members of this class may have boiling points as high as 200 °C.

The solubilities of the individual VOCs in water vary widely, from insoluble to soluble, with many of the oxygenated compounds (ketones and ethers) at the soluble end of the range and the hydrocarbons and substituted hydrocarbons at the insoluble end of the range.

Given that water may be present to varying degrees in such solid materials of environmental significance as soils, solid wastes, and sediments, the water solubility of an individual VOC may in fact control its "solubility" in solid samples.

A.1.2 What is sample preservation?

The sample collection procedures described in EPA analytical methods are designed to ensure that at the time of analysis, the chemical composition of the small volume of material collected from the parent bulk material is representative of the chemical composition of the original material. Considerations regarding sample support and sampling design (discussed in Chapter Nine of the SW-846 manual) ensure that the physical aspects of sample collection (e.g., sample volume and orientation, numbers and distribution of samples) produce data estimates that are representative of the bulk material subject to regulatory decision-making, perhaps millions of gallons a day of discharged wastewater, or thousands of kilograms of solid material. Once collected, a sample should be maintained in a manner that preserves the relationship between it and the bulk material, e.g., the chemical composition of the sample should not change by virtue of being collected. Maintaining that relationship between the sample and the bulk material is referred to as sample preservation.

Several types of sample preservation are employed in EPA methods. The most common method of preservation is to cool the sample to 4 ± 2 °C. Cooling may be applied to many types of sample matrices, including water, soil, sediments, and solid wastes. The temperature of 4 ± 2 °C is used because it represents the temperature at which pure water exhibits its maximum density, hence its minimum volume. However, if aqueous samples are cooled below 0 °C, the water expands significantly as it freezes and may crack the sample container.

By lowering the temperature of the sample, many of the physical, chemical, and biological processes that may cause environmental contaminants to leave the sample (e.g., loss of volatiles to the air) or be transformed into other compounds (e.g., chemical breakdown or biodegradation) are greatly slowed. However, even if the rates of biodegradation are reduced by physical preservation, many environmental matrices of interest contain large numbers of microorganisms that may break down contaminants. Examples include wastewaters from sewage

treatment, surface waters, and surface soil. In these types of matrices, simply reducing the rate at which biodegradation occurs may not be enough to maintain the condition of the original sample.

The most practical way in which to reduce this biological activity in aqueous samples is through the use of chemical preservatives that act as biocides. Historically, this has included preservatives such as sodium bisulfate or hydrochloric acid to adjust the pH for aqueous samples to less than pH 2, at which point, virtually all biological activity ceases.

Adjusting the pH of a solid sample such as a soil, sediment, or solid waste presents a number of other difficulties. In particular, samples containing carbonates should not be acidified due to the potential for effervescence which may result in loss of volatile compounds. Precautions should also be taken when preserving by acidification since certain compounds within the olefins, ketones, esters, ethers, and sulfides classes may react under low pH conditions and possibly not be representative of the material as sampled. Additionally, acidification of solid wastes may evolve toxic gases that may be harmful to field and laboratory personnel. **It is therefore recommended that when collecting wastes of unknown composition, preliminary screening and characterization of potential sample contents should be performed prior to use of acidification as a means to chemically preserve samples designated for determinative analyses.**

Sample collection and preservation procedures should be carefully selected in order to minimize VOC losses prior to sample preparation and determination in the laboratory. Although this guidance discusses some traditional approaches to VOC sample collection and preservation, its main purpose is to provide guidance regarding newer approaches, such as freezing the samples, which may particularly decrease VOC loss in some materials. For additional information regarding the challenges associated with collecting and handling VOC samples, recommended reading includes the "Standard Guide for Sampling Waste and Solids for Volatile Organic Compounds" (ASTM D 4547-98), published by the American Society for Testing and Materials (ASTM). (Ref. 15)

Currently, **it is recommended that VOC solid samples are to be collected, while maintaining a closed-system approach to prevent constituent losses, using an appropriate coring device and immediately transferred to the VOA vial to be used for analysis and should be stored for no longer than 48 hours at $4 \pm 2^{\circ}\text{C}$ prior to analysis or preservation.** Longer storage times at $4 \pm 2^{\circ}\text{C}$ may be appropriate if it can be demonstrated that the VOC concentrations are not adversely affected or that the data generated at the time of sample analysis meets the project-specific data quality objectives. Extended sample storage, up to 14 days from sample collection, may be obtained by either physical or chemical preservation techniques as noted in this Appendix guidance. These preservation techniques can be initiated at the time of sample collection or after arrival in a laboratory. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A.1.3 Do all VOA samples need to be chemically preserved?

No. Only samples that contain analytes that are subject to biological degradation prior to analysis need to be preserved. Samples where aromatic hydrocarbons are target analytes, which are most subject to biological degradation, need to be preserved, unless they are to be analyzed immediately on-site, even if other VOA compound classes are present. Preservation may be inappropriate for highly reactive compounds, e.g., styrene, vinyl chloride, since it may accelerate loss by polymerization or other rapid chemical reaction. Samples for which chlorinated aliphatic hydrocarbons are the only target analytes generally do not need to be preserved. However, all aqueous samples containing free chlorine must be preserved with a dechlorinating agent in order to prevent formation of trihalomethanes and other possible chemical reactions.

A.1.4 Who is the intended audience for this Appendix?

VOCs are frequently Resource Conservation and Recovery Act (RCRA) Program analytes of concern, and thus waste management decisions are often based on characterization of the VOC levels. The intended users of this Appendix guidance are those individuals involved in any way in the collection and preparation of samples for VOC analysis during the characterization of solid materials under RCRA. This may include:

- field sampling personnel
- laboratory analysts
- environmental project managers, whether at a facility regulated under RCRA, or working for a regulatory agency
- Federal, state, and local regulators with oversight responsibilities for sample collection activities
- quality assurance personnel
- data quality assessors.

A.1.5 What does this guidance *not* cover?

This Appendix does *not* provide detailed guidance regarding sampling design or the actual steps in sample preparation and VOC determination in the laboratory. For such guidance, users of this manual should refer to Chapter Nine of SW-846 and the preparation and determinative methods that are selected for analysis as part of the planning process in order to meet the intended data quality objectives.

A.1.6 What equipment is needed?

The site-specific Sampling and Analysis Plan should clearly list the required sample collection equipment necessary to ensure that the loss of volatile constituents will be minimized during the sample collection process. As with all environmental sampling applications, the analytical data usability and representativeness will be affected by improper sample collection techniques. Sampling personnel will be responsible for ensuring that VOA vials are sealed properly using a septum of sufficient thickness without any punctures. The improper vial sealing (i.e., due to excess sample retained on the vial threads) and tightening of caps are the primary factors in the loss of volatiles due to sample collection activities. Care should also be exercised in the selection of approved pre-cleaned and certified VOA vials absent of burrs on the glass. Procedures should be in place for the selection and appropriate use of sample collection devices (i.e., bailer, coring tool, etc.) along with the required decontamination measures. It is also recommended to store one trip blank per cooler when collecting volatile samples in order to assess possible field induced contamination.

A.1.7 How is the guidance organized?

This Appendix is organized as follows:

Section A.2.0 - Project Planning -- Provides an overview of the data quality objectives (DQOs) process as related to the suggested project planning activities prior to sample collection.

Section A.3.0 - Aqueous Sample Matrices and Volatile Organic Compounds – Outlines the appropriate sampling and preservation strategy for aqueous sample matrices.

Section A.4.0 - Solid Materials/Cohesive Soils and Volatile Organic Compounds --
Describes the two most common mechanisms (volatilization and biodegradation) for potential VOC losses during the sample collection process.

Section A.5.0 - History of Practices in the Sampling and Preparation of Solid Materials for VOC Analysis – Provides a summary of the common historical VOC loss mechanisms and discusses the improvements and new developments in sample collection techniques.

Section A.6.0 - Overview of Vapor Partitioning and Methanol Extraction Technologies – Discusses the two most commonly used methods for the laboratory preparation of soils for VOC analysis.

Section A.7.0 - Sample Collection – Describes the sample collection and storage process for various solid matrices.

Section A.8.0 - Approaches to Sample Preparation -- Provides examples of several sample preparation techniques that may be appropriate based on the intended use of the data.

Section A.9.0 - Summary of Findings – Lists the key highlights as discussed in Sections A.2.0 through A.8.0.

Section A.10.0 - References

The EPA requires that a systematic planning process such as, but not limited to, the Data Quality Objectives (DQOs) Process be used for all EPA environmental data collection activities. Systematic Planning is necessary to define the type, quantity, and quality of data a decision maker needs before collecting or generating environmental data. As part of the DQO process, questions such as "what are the possible sample matrices?," "why is the sample being collected?," and "what are the appropriate analytical methods?" can be answered based on the intended use of the data. The Systematic Planning process should also include the preparation of a Quality Assurance Project Plan (QAPP) along with a site-specific Sampling and Analysis Plan (SAP) prior to any sample collection activities. Refer to *Guidance for the Data Quality Objectives Process* (G-4) (August 2000, EPA/600/R-96/055), *Guidance for Quality Assurance Project Plans* (G-5) (February 1998, EPA/600/R-98/018) and Chapter Nine of SW-846 for guidance on how to perform the DQO process and planning guidance associated with RCRA waste sampling and analysis.

During the project planning period it is important to stress to all interested parties that any samples identified as a result of the planning process must be representative of the material subject to investigation, and that each sample handling activity can affect sample integrity and representativeness up through analysis (e.g., VOCs can be lost if samples are not appropriately collected and preserved [See Sec. A.1.3]).

The EPA encourages the use of a performance-based measurement system (PBMS) during selection of sample collection and preparation approaches. The EPA defines PBMS as "a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified, and serve as criteria for selecting appropriate methods to meet those needs in a cost effective manner." The PBMS process permits the use of any appropriate method that demonstrates the ability to meet established criteria while complying with specified data quality needs. In addition, analysts must generate initial and continuing method performance data that demonstrate that the selected approaches were appropriate. Implementation of PBMS does not negate the need for or use of standard or consensus methods. It only eliminates the mandate that they be used exclusively. The following are typical items that should be considered during selection of approaches to VOC sample collection and preservation:

1. VOC concentration range.
2. VOC constituents of interest.
3. Physical characteristics of material, i.e., water content and particle size distribution.
4. Chemical and biological characteristics of material, i.e., acid/base properties, chlorine residual, carbonate content, and microbial activity.
5. Compatibility with selected preparation method.
6. Holding time constraints.
7. Data quality requirements.

All environmental aqueous samples are physically preserved at $4 \pm 2^\circ\text{C}$ immediately after collection in order to improve the overall VOC stability prior to analysis. This preservation process alone has been shown to be effective in preventing the degradation of most constituents for up to seven days from the sample collection date. Depending on the project required VOC constituents, an aqueous sample stability or holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid. The chemical preservatives act as acidifying agents to lower the sample pH and thereby inhibit microbial activity which may cause biological degradation of aromatic hydrocarbons. However, since reactive compounds such as 2-chloroethyl vinyl ether are unstable at low pHs, if these analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. In addition, aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs 48,49) (NOTE: if the aromatic constituents such as benzene, toluene, ethylbenzene, and xylenes (BTEX) are among the analytes of interest, acidification is required for biologically active samples because it has been demonstrated that losses can occur within four hours of sample collection).

The presence of free chlorine in aqueous samples must be monitored and controlled in order to prevent the possible formation of trihalomethanes and reaction with certain compounds such as styrene after sample collection. Therefore, samples containing residual chlorine should be treated with a 10% sodium thiosulfate solution or ascorbic acid prior to acidification in order to reduce the chlorine to unreactive chloride.

Details of procedures and protocols for sample collection must be identified in an approved sampling plan. Aqueous samples for volatile constituents should be collected in vials or containers specifically designed to prevent loss of analytes. In most cases, containers should be provided by the laboratory conducting the analysis. If chemical preservation is required and the laboratory has not pre-preserved the containers, add the appropriate preservative prior to sample collection. Store empty VOC containers on ice in order to reduce potential volatilization while they are being filled. During the sample collection process do not rinse the container before filling and take care to minimize sample overflow that may dilute the preservative. The container should be filled until the water sample forms a positive meniscus at the brim. At this point the container is capped immediately to prevent bubbles and headspace. After the sample has been collected and the container capped, the formation of bubbles can be verified by inverting and lightly tapping the side of the container. Sometimes it is not possible to collect a sample without air bubbles, particularly if the water is aerated. In these cases, the field personnel should record the problem and account for the probable cause. (NOTE: dechlorinating agents should not be mixed with the acid preservative prior to sample collection).

During transport and prior to analysis, samples should be stored in a cooler or refrigerator maintained at $4 \pm 2^\circ\text{C}$ and care should be taken to prevent freezing of the sample and possible container breakage. The sampling plan should indicate how sample shipment will occur along with method of packaging, shipping, and the time schedule relative to sample collection and analytical holding times. Refer to Table A.1 for a summary of the recommended preservation techniques and analytical holding times.

A large number of water VOC sample holding time and stability studies have been performed to determine the degree of degradation which may occur at a variety of concentrations, preservation, and storage conditions. Data from these studies have been reviewed by the Oak Ridge National Laboratory (ORNL) in order to develop an approach for assessing the data

confidence from analyses completed beyond the regulatory holding time of 14 days. This approach is based on methodology, referred to as "Practical Reporting Times," that were developed by ORNL in the early 1990's, and described in a summary report listed in Ref. 47. Users may find the data provided in Tables 2 and 3 of this referenced report to be helpful in estimating the post-holding time degradation of VOCs in water and for determining the potential data impact from analyses completed beyond the required holding time. However, the user is cautioned that the holding times provided in this report are estimations based on actual analytical data, and the true values are relative to the on-site sample matrix conditions. See the footnote following Table A.1 regarding holding time extensions.

A.3.1 Alternative Considerations for Sample Holding Time Criteria

Recognition that holding times for environmental contaminants are analyte-specific and highly variable is not new. (Refs. 52,53,54). Environmental contaminants may be short-lived, destroyed by preservation, or highly resistant to degradation. Understanding and applying historical knowledge (Table A.1) can be important and valuable. (Ref. 55) Therefore, we encourage consideration of alternative holding times for several reasons:

1. Project planning,
2. Performance based data review processes,
3. Analytical method selection,
4. Streamlined verification of unexpected or suspect analytical results, and
5. Design of alternative quality control procedures.

Specific examples of how to implement the information incorporated in Table A.1 include the following: During project/systematic planning, field measurement or quick-turn-around analyses must be identified as critical if particular contaminants of concern for a project are easily lost or destroyed. Currently, data review guidelines suggest samples analyzed within 2 weeks of collection be accepted as uniformly reliable, and analyses completed >2 weeks after sample collection are uniformly assessed as unacceptably uncertain. This review judgement is not technically defensible. Many of the most common contaminant decision drivers listed in Table A.1 are important, because they are stable over time, e.g., chlorinated solvents. For these contaminants, cooperative Inter-Agency research has demonstrated no significant change in results from analyses performed at 30 days, often as long as 96 days, after collection and preservation. *NOTE: this extension assumes preservation of samples as identified in Table A.1.* In addition, longer holding times than those specified in Table A.1 may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

The resistance to degradation of these frequent environmental drivers offers additional process improvement opportunities. Utilization of a second VOA sample analyzed beyond the recommended holding time is a mechanism to verify or independently determine unexpected results or correct laboratory errors that cannot be addressed within the current 2-week window. With no significant loss of confidence in the results, this approach eliminates the schedule delays and expense of sampling crew mobilization.

In addition, the use of site-specific performance evaluation material is recognized as a high confidence mechanism to ensure reliability of project data. However, the historical perception of short shelf-life for volatile organics in water eliminates implementation of this approach as a viable

quality control/quality assurance system component for water monitoring programs. Table1 and the associated references contain documentation of appropriate analytes and procedures to develop and implement these alternatives.

Table A.1
Recommended VOC Sample Preservation Techniques and Holding Times

Sample Matrix	Preservative	Holding Time	Comment
Aqueous Samples With No Residual Chlorine Present	Cool to $4 \pm 2^{\circ}\text{C}$.	7 days	If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples With No Residual Chlorine Present	Cool to $4 \pm 2^{\circ}\text{C}$ and adjust pH to less than 2 with HCl or solid NaHSO_4 .	14 days ¹	Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
Aqueous Samples With Residual Chlorine Present	Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to $4 \pm 2^{\circ}\text{C}$.	7 days	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. If MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary and the holding time is extended to 14 days.
Aqueous Samples With Residual Chlorine Present	Collect sample in a pre-preserved container containing either 25 mg ascorbic acid or 3 mg of sodium thiosulfate per 40-mL of chlorinated sample volume containing less than 5 mg/L of residual chlorine. Cool to $4 \pm 2^{\circ}\text{C}$ and adjust pH to less than 2 with HCl or solid NaHSO_4 .	14 days ¹	Samples containing greater than 5 mg/L of residual chlorine may require additional amounts of dechlorinating agents. Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible. Caution: never add acid preservative directly to a dechlorinating agent prior to sample collection.
Solid Samples ²	Sample is extruded into an empty sealed vial and frozen on-site to $< -7^{\circ}\text{C}$.	14 days ¹	Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Table A.1 (Continued)

Sample Matrix	Preservative	Holding Time ¹	Comment
Solid Samples ²	Sample is extruded into an empty sealed vial and cooled to $4 \pm 2^\circ\text{C}$ for no more than 48 hours then frozen to $< -7^\circ\text{C}$ upon laboratory receipt.	14 days ¹	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into an empty sealed vial and cooled to $4 \pm 2^\circ\text{C}$ for no more than 48 hours then preserved with methanol upon laboratory receipt.	14 days ¹	Analysis must be completed within 48 hours if samples are not preserved with methanol prior to the expiration of the 48 hour period.
	Sample is extruded into an empty sealed vial and cooled to $4 \pm 2^\circ\text{C}$.	48 hours	
	Cool to $4 \pm 2^\circ\text{C}$ the coring tool used as a transport device	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to $< -7^\circ\text{C}$ or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing.
	Freeze to $< -7^\circ\text{C}$ the coring tool used as a transport device	48 hours	The holding time may be extended to 14 days if the sample is extruded to a sealed vial and either frozen to $< -7^\circ\text{C}$ or chemically preserved. Coring tools should not be frozen below -20°C due to potential problems with tool seals and the loss of constituents upon sample thawing.
	Sample is extruded into a vial containing reagent water and frozen on-site to $< -7^\circ\text{C}$.	14 days ¹	Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.
	Sample is extruded into a vial containing reagent water and cooled to $4 \pm 2^\circ\text{C}$ for 48 hours or less then frozen to $< -7^\circ\text{C}$ upon laboratory receipt.	14 days ¹	Analysis must be completed within 48 hours if samples are not frozen prior to the expiration of the 48 hour period. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Table A.1 (Continued)

Sample Matrix	Preservative	Holding Time ¹	Comment
Solid Samples ²	Sample is extruded into a vial containing reagent water and 1 g NaHSO ₄ and cooled to 4 ± 2°C.	14 days ¹	Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.
	Sample is extruded into a vial containing methanol and cooled to 4 ± 2°C.	14 days ¹	Additional methanol extract storage time beyond 14 days may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

¹ A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from preservation, storage and analyses performed outside the recommended holding times.

² For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

During the selection of VOC sample collection and preservation approaches, it is important to understand the mechanisms of VOC loss inherent to solid materials and VOCs. In general, uncontrolled losses occur through both volatilization and biodegradation. However, for some compounds, e.g., vinyl chloride, acrylonitrile, 2-chloroethylvinyl ether, and styrene, rapid losses can occur through chemical reaction, as well. (Ref. 46)

In most solid materials, VOCs coexist in gaseous and liquid phases, as well as sorbed to the solid particles. The molecular diffusion coefficients of VOCs in the gaseous phase are high enough to allow for the immediate volatilization of those VOCs from a freshly exposed sample surface, resulting in a loss to the surrounding atmosphere. If the sample matrix is porous, these losses will continue as VOCs below the surface diffuse outward. Furthermore, once the gaseous phase is lost, the dynamic equilibrium between the gaseous phase and the liquid and sorbed VOC phases will result in rapid transformations of the liquid and sorbed VOCs to the gaseous phase, where they can continue to be lost to the atmosphere. (Ref. 4) Thus, the primary goal of preservation is to minimize or eliminate the loss of the compounds of concern through direct volatilization to the atmosphere.

The biodegradation of VOCs usually involves compound loss by biological processes mediated by naturally-occurring micro- and macro-organisms found in solid environmental samples such as soils and sediments. Aerobic processes are often of greatest concern, but anaerobic organisms in some sediments and soils can also result in significant losses of VOCs. Biodegradation may be of concern in waste samples, particularly those that may have been stored outdoors.

Most soil sample collection procedures involve intrusive sampling operations that can create or enhance aerobic conditions within a sample. Aerobic conditions can occur by disaggregation of the particles in the solid, or by simple exposure of the sample to air (e.g., collection of a sediment sample from under standing water). Soil samples should be collected immediately or as soon as practical after exposure of the soil during such activities as tank removal or excavation in order to minimize VOC losses from uncontrolled aerobic processes. Unless precautions as noted in this Appendix are employed, aerobic conditions will then persist during handling and storage of the sample.

The rate of biodegradation is dependent on several factors, including the indigenous microbes, the chemical properties of the individual VOC, the total VOC concentration, the chemical properties of the solid matrix, and temperature. In general, the biodegradation mechanism for soil VOCs is not as large a source of determinate error as volatilization. Volatilization losses of an order of magnitude can occur in minutes to hours, whereas losses of a similar magnitude due to biodegradation usually take days to weeks.

Biodegradation is compound selective whereby, under aerobic conditions, the biological mechanisms favor the degradation of aromatic hydrocarbons over the loss of halogenated (chlorinated) hydrocarbons. (Refs. 1,2,4) Aromatic hydrocarbons such as benzene, toluene, ethyl benzene, and xylenes (collectively referred to as BTEX) can be lost in days from samples stored at $4 \pm 2^\circ \text{C}$, while losses of chlorinated hydrocarbons by biodegradation over the same period can be relatively insignificant. Major benzene and toluene biodegradation losses (50% or more) have been observed when soils are stored at room temperature (22°C) for five (5) days and near complete concentration reduction when stored for fourteen (14) days at 4°C . (Refs. 1,2,4,6,11,17) For extremely biologically active soils this can occur in less than five days. (Ref. 11)

Due to the above mechanisms, attempts are made from the beginning to maintain sample integrity and representativeness. In doing so, approaches often use various combinations of chemical (e.g., methanol) and physical (e.g., freezing) preservation procedures and collection (e.g., single transfer to air-tight vial) and storage practices (e.g., holding times) to minimize VOC loss. Some of these approaches are presented within this guidance.

A.5.0 HISTORY OF PRACTICES IN THE SAMPLING AND PREPARATION OF SOLID MATERIALS FOR VOC ANALYSIS

A.5.1 Traditional Practices

Over the past 20 years, solid samples obtained for VOC analysis were collected using a spatula -type device to completely fill a container for transfer off-site before the introduction of certain preparation steps and analysis within a 14-day holding time. VOC sampling procedures recommended the use of clean stainless steel utensils to completely fill either 40-mL to 250-mL glass containers. The containers were then closed with polytetrafluoroethylene (PTFE)-lined caps. Sample containers were stored in coolers at $4 \pm 2^\circ \text{C}$ and shipped to field or off-site support laboratories for subsampling (usually with 1 to 5 g aliquots) and subsequent analysis. The common holding time for these bulk soil samples, held at $4 \pm 2^\circ \text{C}$, was 14 days.

During the 1990s, research efforts demonstrated that the above VOC bulk sampling procedure is inaccurate and produces VOC results that are biased low. (Refs. 3,8,10,16,30,31,32,33,34) The studies showed that bulk samples can lose 90% or more of their VOC content prior to analytical measurement. (Refs. 3,8,10,16,29,31,32,33) Reasons identified for these losses include:

1. Volatilization from exposure of the solid surface near the time of collection. (Refs. 3,8)
2. Volatilization from intermediate storage containers (e.g., core barrel liners, plastic bags, etc.). (Refs. 4,10,13,17,30)
3. Volatilization from disaggregation of the solid during collection. (Refs. 3,8)
4. Volatilization from failed seals on the PTFE-lined caps of the bottles or volatile organic analyte (VOA) vials (can be caused by soiling of cap and bottle ring closures during filling of containers). (Refs. 3,8)
5. Volatilization during laboratory subsampling of the bulk samples. (Refs. 3,8)
6. Biodegradation (principally of aromatic hydrocarbons, especially benzene and toluene) during storage (probably hastened by disaggregation of soils during sampling). (Refs. 3,8,11)
7. Reaction of chemically reactive compounds during sample storage.
8. Pressure changes during sample collection and transport.

A.5.2 Improvement of Sample Collection Techniques

Due to concerns regarding the loss of VOCs, particularly in samples containing low concentrations of VOCs ($<200 \mu\text{g/kg}$) during traditional sampling practices, the scientific community investigated other approaches to VOC sample collection and preparation. A closed-system purge-and-trap technique was developed and tested for the analysis of low-level concentrations of VOCs in solids. The methanol extraction option for high concentrations ($>200 \mu\text{g/kg}$) and oily wastes remained unchanged. The Office of Solid Waste promulgated Method 5035 as part of Update III to the Third Edition of SW-846 on June 13, 1997. (Ref. 38) As an active participant in these studies in conjunction with OSW, the American Society for Testing and Materials (ASTM) published the

"Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds" (ASTM D 4547-98). (Ref. 15)

These documents include the immediate in-field transfer of the sample (by a coring tool of 2 to 5 g capacity) into a tared VOA vial (of 22 to 40 mL capacity) that contains acidified reagent water (most often acidified by 1g NaHSO₄ per 5g of soil) so that a vapor partitioning preparation procedure (see Sec. A.6.0 of this Appendix) can be performed by the laboratory on the sample without reopening the vial. A second in-field transfer to a tared VOA vial containing 5 to 10 mLs of methanol is used for VOC soil concentrations larger than 200 µg/kg.

Another technique described is the immediate in-field collection and maximum 48 hour storage in an air-tight coring device/container (such as the EnCore™ sampler) so that the laboratory preservation and preparation procedures described for the closed-system purge-and-trap (Method 5035) or headspace (Method 5021) can be performed. (Ref. 39) (An EnCore™ sampler is a device that can be used for both sample collection and as the sample transport and storage device. See Sec. A.8.0)

Both documents recommend similar approaches to sample preservation and preparation in order to minimize VOC loss and address the collection of cohesive solids whereby a coring tool collects a relatively undisturbed sample by compression, and then extrudes the sample into an appropriate VOA vial. The documents also provide guidance for the collection of cemented materials and non-cohesive materials (e.g., dry sand, mixtures of gravel and fines) and collectively address factors that must be considered when selecting the most appropriate approach for VOC sample preservation and preparation, including expected concentrations of VOCs (high versus low). A screening method for determining whether a sample contains high or low concentrations of VOCs (Method 3815) is available for making these determinations on-site. (Ref. 40)

A.5.3 New Developments

Since the publication of the new VOA sampling techniques for solids, the scientific community has continued to investigate additional techniques to further improve sample collection and preservation to minimize VOC loss. For example, studies were conducted regarding the freezing of samples without the use of chemical preservatives (see Sec. A.8.0), use of "empty VOA vials," and more information was gained regarding acidification of samples, as discussed below. (Refs. 4,19)

Current practice recommends the use of NaHSO₄ to acidify reagent water in VOA vials prior to addition of the sample when preservation is necessary. (Ref.1) This acidification is one means used to minimize loss of VOC due to biodegradation. However, acidification is not recommended for solids or aqueous samples with significant levels of carbonates, because the acidification can cause effervescence and the loss of VOCs. In 1998 and 1999, other adverse effects of acid preservation of soils were discovered i.e., chemical breakdown of certain classes of compounds. Additionally, certain VOC components such as 2-chloroethylvinyl ether are lost by the acidification. An artifact is sometimes observed for acetone in that acidification of certain soils may cause the formation of acetone. (Refs. 4,37)

The approaches recommended in Sec. A.8.0 of this guidance incorporate the new developments in solid sample preparation for VOC analysis.

A.6.0 OVERVIEW OF VAPOR PARTITIONING AND METHANOL EXTRACTION TECHNOLOGIES

Vapor partitioning and methanol extraction are the two most commonly used methods for the laboratory preparation of soils for VOC analysis. This section briefly discusses these two procedures, and their relative advantages and disadvantages. For further information, ASTM D 4547-98 (Ref. 15) discusses the merits of vapor partitioning relative to the use of methanol extraction; and Method 5035 relates concerns regarding the use of methanol.

Selection of the preparation technology should be made during the systematic planning process prior to sample collection given that the selection will dictate subsequent sample collection and preservation practices. One technology may be preferred based on the project data quality objectives and target analytes, and the sample collection and handling approaches need to be compatible with the chosen technology.

Each preparation technology involves use of a VOA vial for sample collection and transport. Approaches for preparation of the vials (with and without preservatives), often based on the technology to be used, will be discussed in a section to follow.

A.6.1 Vapor Partitioning

One means of vapor partitioning involves the direct analysis of a sample by purge-and-trap (Method 5035). This technique is routinely used for the analysis of volatiles in environmental samples and is considered more sensitive than the headspace technique. By purging samples at higher temperatures, higher molecular weight compounds can be detected. However, the purge-and-trap technique requires more time for sample preparation.

Another means of vapor partitioning involves the direct analysis of a sample by equilibrium headspace (Method 5021). This technique is most suited for the analysis of very light molecular weight volatiles in samples that can be efficiently partitioned into the headspace gas volume from the liquid or solid matrix sample. Higher boiling point volatiles are not detected with this technique due to their low partition rate in the gas headspace volume. In addition, the technique is generally less sensitive than purge-and-trap, however, it is preferred for the analysis of gases, highly water-soluble compounds, and very light molecular weight volatiles which may not be analyzed using the purge-and-trap technique.

For both vapor partitioning techniques, the vapor is removed for analysis without opening the container. Heat and water are usually used to assist in the direct partitioning of VOCs from the solid matrix. Vapor partitioning is applicable to VOC soil concentrations of 2 to 200 ppb. Methods 5021 or 5035 commonly require 2- to 5-g soil aliquots collected in individual 20- to 40-mL VOA vials, depending on the specific instrumentation used in the selected purge-and-trap or headspace method. Only one analysis per VOA vial can be done using purge-and-trap or headspace (Methods 5035 or 5021).

Vapor partitioning can offer lower detection limits than methanol extraction because no dilution is involved. In addition, there are no organic solvent interferences and no use of regulated organic solvents (e.g., methanol), which requires special handling and disposal practices. Use of methanol may generate a flammable waste that is hazardous based on the ignitability characteristic (40 CFR § 261.21) or a listed waste (40 CFR § 261, Appendix VII).

A.6.2 Methanol Extraction

Methanol extraction involves the extraction of VOCs from a sample with methanol, and the subsequent transfer of an aliquot of the extract to water (dilution) for either purge-and-trap or headspace analysis. After extraction with methanol (anywhere from 1:1 methanol to soil to a 10:1 methanol to soil ratio); the extract typically receives a 50-fold dilution. Methanol extracts must be diluted to minimize adverse effects of methanol on analytical instrumentation. However, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report results for samples containing significant moisture contents on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations.

One advantage of a methanol extract is it may be tested more than once. Methanol extracts of soil are applicable to a wide range of high to low concentrations, e.g., 50 ppb to several ppm. Once a methanol extract is obtained, it can be stored at $4 \pm 2^{\circ}\text{C}$ for two weeks, and sufficient volume is present for multiple VOC determinations. Additional extract storage time beyond two weeks may be acceptable if the desired VOC constituent stability can be demonstrated from appropriate performance data.

As noted above, concerns exist regarding the use of methanol extraction. The information to follow provides recent observations regarding the use of methanol for VOC analysis.

A.6.2.1 Contact Time Effect

Methanol extraction can provide more robust, larger or accurate values for VOCs when compared to vapor partitioning results. (Refs. 5,9,16,27,29,30,32,33,41,42) However, methanol extract results tend to increase with time as the sample contact time increases. (Refs 27,33) State agencies implementing methanol extraction for soil VOCs often require either a minimum contact time of one day, or the soil is to be sonicated for 20 to 30 minutes at 40°C with the methanol prior to analytical measurement of VOCs. The actual contact time should be sufficient enough to efficiently extract all VOC constituents of interest and to allow for the complete breakdown of agglomerated solid materials.

Particularly volatile VOCs (e.g., benzene, dichloroethene) in sandy soils are not expected to show this effect of contact time. The less volatile VOCs (e.g., xylenes) in an organic rich soil or clay can be expected to demonstrate higher results with increased contact time. (Refs. 5,9,27,33)

A.6.2.2 Safety and Hazardous Waste Generation Concerns

A primary disadvantage of methanol extraction is that it poses hazards to personnel due to its toxicity and flammability. Finally, the addition of methanol to a sample is likely to cause the sample to fail the ignitability characteristic or to become a listed waste, thereby making the unused sample volume a hazardous waste.

A.7.0 SAMPLE COLLECTION

A.7.1 Collection of Samples for Analysis

After a fresh surface of the solid material is exposed to the atmosphere, the subsample collection process should be completed in the least amount of time in order to minimize the loss of VOCs due to volatilization. Removing a subsample from a material should be done with the least amount of disruption (disaggregation) as possible. Additionally, rough trimming of the sampling location's surface layers should be considered if the material may have already lost VOCs (been exposed for more than a couple of minutes) or if it may be contaminated by other waste, different soil strata, or vegetation. Removal of surface layers can be accomplished by scraping the surface using a clean spatula, scoop, knife, or shovel. (Refs. 15,51)

A.7.1.1 Subsampling of Cohesive Granular but Uncemented Materials Using Devices Designed to Obtain a Sample Appropriate for Analysis

Subsamples of the appropriate size for analysis should be collected using a metal or rigid plastic coring tool. For example, coring tools for the purpose of transferring a subsample can be made from disposable plastic syringes by cutting off the tapered front end and removing the rubber cap from the plunger or can be purchased as either plastic or stainless steel coring devices. These smaller coring devices help to maintain the sample structure during collection and transfer to the VOA vial as do their larger counterparts used to retrieve subsurface materials. When inserting a clean coring tool into a fresh surface for sample collection, air should not be trapped behind the sample. If air is trapped, it could either pass through the sampled material causing VOCs to be lost or cause the sample to be pushed prematurely from the coring tool. The commercially available EasyDraw Syringe™ and Powerstop Handle™ and Terra Core™ sampler coring devices are designed to prevent headspace air above the sample contents. For greater ease in pushing into the solid matrix, the front edge of these tools can be sharpened. The optimum diameter of the coring tool depends on the following: size of the opening on the collection vial or bottle (tool should fit inside mouth), dimensions of the original sample, particle size of the solid materials (e.g., gravel-size particles would require larger samplers), and volume of sample required for analysis. For example when a 5-g subsample of soil is specified, only a single 3-cm³ volume of soil has to be collected (assuming the soil has density of 1.7 g/cm³). Larger subsample masses or more subsample increments are preferred as the heterogeneity of the material increases. After an undisturbed sample has been obtained by pushing the barrel of the coring tool into a freshly exposed surface and then removing the corer once filled, the exterior of the barrel should be quickly wiped with a clean disposable towel. The next step varies, depending on whether the coring device is used for sample storage and transfer or solely for transfer. If the coring tool is used as a storage container, cap the open end after ensuring that the sealing surfaces are cleaned. If the device is to be solely used for collection and not for storage, immediately extrude the sample into a VOA vial or bottle by gently pushing the plunger. The volume of material collected should not cause excessive stress on the coring tool during intrusion into the material, or be so large that the sample easily falls apart during extrusion. Obtaining and transferring a sample should be done rapidly (<10 seconds) to reduce volatilization losses. If the vial or bottle contains methanol or another liquid, it should be held at an angle when extruding the sample into the container to minimize splashing. Just before capping, a visual inspection of the lip and threads of the sample vessel should be made, and any foreign debris should be removed with a clean towel, allowing an airtight seal to form.

A.7.1.2 Devices that Can be Used for Subsampling a Cemented Material

The material requiring sampling may be so hard that even metal coring tools cannot penetrate it. Subsamples of such materials can be collected by fragmenting a larger portion of the material using a clean chisel to generate aggregate(s) of a size that can be placed into a VOA vial or bottle. When transferring the aggregate(s), precautions must be taken to prevent compromising the sealing surfaces and threads of the container. Losses of VOCs by using this procedure are dependent on the location of the contaminant relative to the surface of the material being sampled. Therefore, caution should be taken in the interpretation of the data obtained from materials that fit this description. As a last resort when this task can not be performed onsite, a large sample can be collected in a vapor-tight container and transported to the laboratory for subsampling. Collecting, fragmenting, and adding the sample to a container should be accomplished as quickly as possible.

A.7.1.3 Devices that Can be Used for Subsampling a Non-cohesive Granular Material

As a last resort, gravel, or a mixture of gravel and fines that can not be easily obtained or transferred using coring tools, can be quickly sampled using a stainless steel spatula or scoop. If the collection vial or bottle contains methanol or an aqueous solution, samples should be transferred with minimal splashing and without the spatula or scoop contacting the liquid contents. For some solids, a wide-bottom funnel or similar channeling device may be necessary to facilitate transfer to the container and prevent compromising of the sealing surfaces of the container. Caution should be taken in the interpretation of the data obtained from materials that fit this description. Losses of VOCs are likely because the nature of the sampling method and the noncohesive nature of the material expose more surface area to the atmosphere than other types of samples. During the sampling process, noncohesive materials also allow for the separation of coarser materials from fines, which can skew the concentration data if the different particle sizes, which have different surface areas, are not properly represented in the sample.

A.7.2 Use of the EnCore™ Sampler (or Equivalent) for Sample Transport and Storage

The EnCore™ sampler is a sampling device that can be used as both a simultaneous coring tool for cohesive soils and a transport device to a support laboratory (field or off-site). The EnCore™ sampler is intended to be a combined sampler-storage device for soils until a receiving laboratory can initiate either immediate VOC analysis, or preserve extruded soil aliquots for later VOC analysis. It is meant to be disposed after use. The commercially available device is constructed of an inert composite polymer. It uses a coring/storage chamber to collect either a 5 g or 25 g sample of cohesive soils. It has a press-on cap with hermetically vapor tight seal and locking arm mechanism. It also has a vapor tight plunger for the nondisruptive extrusion of the sample into an appropriate container for VOC analysis of soil.

An individual disposable EnCore™ sampler (or equivalent) is needed for each soil aliquot collected for vapor partitioning or methanol sample preparation. Upon soil sample collection, the EnCore™ sampler is stored at $4 \pm 2^\circ\text{C}$ until laboratory receipt within 48 hours. Upon laboratory receipt, soil aliquots are extruded to appropriate tared and prepared VOA vials.

Validation data have been provided to support use of the EnCore™ sampler for VOC concentrations in soil between 5 and 10 ppm, for two (2) sandy soils, with a 2-day holding time at $4 \pm 2^\circ\text{C}$. Preliminary data (Ref. 25) demonstrate an effective 2-day (48-hour) holding time at $4 \pm$

2°C for three sandy soil types with VOC concentrations at 100 ppb (benzene and toluene at 300 ppb), as well as an effective 1 or 2 week holding time at -12°C (freezing temperature). Recent published work (Ref. 43) neither definitively supports or shows the EnCore™ device to be ineffective for sample storage at these preservation temperatures. Soils stored in the EnCore™ device for 2 calendar days at $4 \pm 2^\circ\text{C}$ are subject to loss of BTEX compounds by biodegradation if the soil is an aerated, biologically active soil (e.g., garden soil) (Ref. 24), but this BTEX loss is eliminated for up to 48 hours under freezing conditions. (Ref. 2)

Further details on the EnCore™ sampler can be found in ASTM D4547-98 (Ref. 15) or other publications.

A.7.3 Concerns Regarding Use of Core Barrel Liners

One geotechnical technique for retrieval of bulk soil from subsurface regions is ring-lined barrel samples. Core barrel liners fit snugly within a corer and can be constructed of steel or brass (which is inert to VOCs). Cylindrical cores of subsurface soil can be obtained anywhere from 1 to 4 inches in diameter of varying lengths in feet.

Core barrel liners have been used as both a sample collection and storage device for VOC soil samples. Upon retrieval with subsurface soil, the core barrel liner (brass) is covered on both ends with a thin sheet of PTFE or with aluminum foil. Plastic caps are pressed over the ends to hold the PTFE/aluminum in place. The core barrel liner sample is maintained at $4 \pm 2^\circ\text{C}$ during shipment and storage at a laboratory. Sample preparation for VOC analysis is initiated by opening the core barrel coverings and sub-sampling the soil with a coring tool for analyses by either the vapor partitioning or methanol extraction options.

Experimental work has demonstrated that the core barrel transport and storage procedure is ineffective for a 2-day storage and holding time. (Refs. 4,10,13,16, 36) PTFE coverings (0.02 mm and 0.05 mm thickness) and aluminum foil will not prevent losses of 30-90% for certain volatile compounds (dichloroethene, benzene and trichloroethene). Therefore, the core barrel liners should be used as sample collection and transfer devices only with the least amount of elapsed time as possible prior to sample preparation.

A.7.4 After Collection -- Sample Handling and Storage

A.7.4.1 Holding Times

Published holding times should be followed, unless performance data can be produced to support longer time periods.

This guidance assumes a 48-hour holding time, unpreserved at $4 \pm 2^\circ\text{C}$, between sample collection and analysis or preservation of VOC soil aliquots in VOA vials. Most validation data provided to support or justify an approach listed the holding time as 48 hours. The 48-hour holding time results for VOC in soil can provide average recoveries of 80% or more. However, recoveries from samples stored for 5 days are less successful. Little data exists on the impact of holding times between 48 hours and 5 days.

Implementing a 48-hour holding time can be difficult when transporting VOC soil samples (via overnight air carrier) from the field to an off-site support laboratory. All interested parties i.e., field and laboratory personnel need to be cognizant that the 48 hour holding time begins **from the time of sample collection**. If the VOC analysis cannot be completed prior to the expiration of the initial 48 hour period, other

preservation measures (i.e., freezing, chemical preservation, and methanol extraction) are required in order to extend the analysis holding time to 14 days from the time of sample collection.

A.7.5 Quality Control

Quality control checks to be employed during field sampling activities should include the collection, preparation, and analysis of the various QC samples discussed below:

Note: The exact specifications and need for the following QC samples should be outlined in the project planning documents.

1. Field duplicate: A field duplicate may be prepared at a frequency of one per day per matrix. The field duplicate is an independent sample which is collected as close as possible to the same point in time and space as the primary field sample. Field duplicates are used to estimate the reproducibility (precision) of the sampling process.
2. Trip blank: Trip blanks should be prepared at a frequency of one per day of sampling during which samples will be collected for VOCs. Trip blanks are prepared using reagent water (see Chapter One for definition) prior to the site visit at the time sample containers and kits are transported to the site. The trip blank will accompany the field samples throughout all sample collection and transport operations. This blank will not be opened during sampling activities and will be used to assess sample contamination originating from sample transport, shipping, or site conditions.
3. Field blank: A field blank conversely is prepared on-site during the sample collection activities using the same reagent water source used to prepare the trip blank. The field blank should be collected and preserved in the same manner as the environmental samples. The results from this analysis are used to assess sample contamination originating predominantly from field sampling conditions.
4. Equipment rinsate: An equipment rinsate blank should be collected from sample collection devices used for each distinct sample matrix. The equipment blanks are obtained either prior to or during sample collection activities. The results from these analyses are used to assess possible sample contamination from sampling equipment.
5. Temperature blank: A temperature blank prepared with a water-filled vial or a suitable thermometer, should be included with each cooler of samples designated for transport. Upon sample receipt, the laboratory will use the temperature blank or thermometer to determine the internal temperature of each cooler. Acceptable temperatures are $4 \pm 2^{\circ}\text{C}$ for refrigerated aqueous and solid samples and $< -7^{\circ}\text{C}$ for frozen solid samples.
6. Matrix spike and matrix spike duplicate: Additional sample aliquots should be collected when matrix spike and matrix spike duplicate analyses are required. Matrix spikes are aliquots of environmental samples to which known concentrations of certain target analytes have been added before sample manipulation from the preparation, cleanup, and determinative procedures have been implemented. The matrix spike analysis is used to assess the performance of the method by measuring the effects of interferences caused by

the sample matrix and reflects the accuracy of the method for the particular matrix in question.

7.6 Interferences / Artifacts of Analysis

When aqueous and solid samples are acidified it can lead to losses of highly reactive compounds such as 2-chloroethylvinyl ether through chemical reaction. Additionally, acidification of certain soils with sodium bisulfate may produce a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) Furthermore, *meta*- and *para*-xylene co-elute on most analytical columns and need to be reported as an isomeric pair. Acid preservation of samples to be analyzed for methyl *tert*-butyl ether (MTBE) should be avoided because use of a high temperature sample preparation method (Methods 5021, 5030, or 5032) can cause degradation of the MTBE to *tert*-butyl alcohol (TBA) during the high temperature sample preparation step. (Refs. 49,50)

Since aqueous samples containing residual chlorine must be dechlorinated to prevent the formation of trihalomethanes and other chlorinated compounds, the sample should be added to the dechlorinating agent prior to acid preservation. The addition of sodium thiosulfate to an acidified sample will generate sulfur dioxide which may interfere with the determination of gaseous VOC constituents of interest.

The project chemist should research and review historical data pertaining to the use of VOCs at the site under investigation. If previous data indicates that tetrachloroethylene or trichloroethylene were used at the site and their daughter products dichloroethene and dichloroethane are present, then vinyl chloride may also be present. In this scenario acid preservation would not be appropriate due to the reactive nature of vinyl chloride.

If the sampling location is known to contain polymers that were manufactured from monomers, then both vinyl chloride or styrene could be present. For this situation, due to the potential for reactive compounds present, acid preservation would not be necessary.

Pre-testing of a representative soil sample, prior to collection, with acid or bisulfate may show effervescence if carbonaceous materials are present. If bubbling occurs during chemical preservation, samples should not be collected with acid or bisulfate preservative. If the soil sample is a loamy material or contains a high proportion of decayed matter then acid preservation may generate acetone as a byproduct. The sampling personnel should examine and pre-test the soils to be collected prior to actual collection in order to make the proper determination for the correct preservation technique.

The laboratory should fully document whenever sample matrix interferences are suspected and can be attributed to poor analytical method quality control data. It is also important for the laboratory area where volatile analyses are performed to be completely free of solvents. Special precautions must be taken for the analysis of methylene chloride, since random background levels will result if the analytical and storage areas are not isolated from all sources of atmospheric methylene chloride.

This section provides examples of approaches to sample preparation that include prepared vials (e.g., chemical preservation approaches) and use of empty vials (other means such as freezing used for preservation). Complete validation data is not available for all approaches. Analysts are responsible for showing that any given approach is appropriate for the intended use of the data.

Typically, as part of these procedures, a cohesive soil subsample (2 to 5g) from a freshly exposed sampling trench, geotechnical coring device/probe, etc., using a coring tool such as a cut-off syringe or purchased device (e.g., EasyDraw Syringe™ and Powerstop Handle™ or EnCore™), is extruded immediately to either a tared empty VOA vial or to a tared prepared VOA vial. Precautions with handling tared vials i.e., not applying additional labels, markings, and seals are necessary to ensure an accurate sample weight. Once filled with sample, the VOA vials are then capped (with PTFE-lined septa) until VOC sample preparation. Three or more replicate VOA vials (e.g., two for vapor partitioning and additional ones for any matrix spike QC analysis) are utilized by either technique, as well as one more soil aliquot for a percent moisture determination. One coring tool (disposable or reusable) can be used at each soil sampling location by providing co-located cores for the replicate VOA vials. The same coring tool can be used to collect an additional co-located soil for the percent moisture determination typically required by the laboratory preparation procedures. If the coring tool can be properly capped to prevent moisture loss, the coring tool can be used as a storage container for percent moisture. Note: should freezing be used as a means to preserve samples in the field, the aliquot reserved for percent moisture determination should not be frozen.

The preparation of samples for VOC analysis can be initiated either in the field at the time of collection using the prepared VOA vials, or at either an on- or off-site support laboratory using either the empty VOA vials (note the manual puncture of septa to introduce reagent water prior to analysis is not recommended) or a coring tool (e.g., the EnCore™ sampler) that can also serve as a sample transport device. A separate EnCore™ sampler is required for each replicate VOA vial used for VOC analysis.

When determining VOCs over the complete concentration range of ppb to several ppm, four (4) or more VOA vials may be required for each sampling point. For example, at least one VOA vial is necessary for methanol extraction when selected to analyze high VOC concentrations, while at least two vials are necessary for when vapor partitioning is to be used because low VOC concentrations (<200 ppb) are expected. A fourth VOA vial may be necessary for percent moisture determination so that VOC concentrations can be corrected for moisture content and/or methanol dilution factor, if required. A set of replicates for a single investigative soil sample are often composed of the following:

1. Two (2) 40-mL VOA vials for direct vapor partitioning measurement. These are needed for the most sensitive measurements - one is kept in reserve for any necessary repeat analysis. The upper concentration value of the vapor partitioning method's calibration range limits the usability of these direct measurements.
2. One (1) 40-mL VOA vial for methanol extraction of soil aliquot prior to vapor partitioning. Once a methanol extract is obtained, an aliquot of this extract is diluted fifty-fold (50) or more with water and is tested by vapor partitioning as a water matrix. The 50-fold dilution is necessary to minimize interferences in vapor partitioning measurements of water matrices. Methanol extracts have no

upper limit of measured VOC concentration since the extract can be sub-aliquoted for different dilutions.

3. One (1) 60-mL VOA vial for any percent moisture determination to report VOC results on a moisture corrected basis, if necessary. Also note that solid samples with a significant moisture content ($>10\%$) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. (Ref. 51) The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Therefore, in order to report this type of sample result on an "as received" basis, the detected concentration needs to be corrected by the solvent/water dilution factor. See Method 8000 for an explanation and the applicable calculations. The physical preservation ($4 \pm 2^\circ\text{C}$) of this vial is not as critical as for the VOC analytes in soil.
4. VOA vials for any QC audits such as duplicates, matrix spikes, etc.

Please note that a VOA vial should always be collected for methanol extraction unless it is known in advance that VOCs will not exceed the upper usable concentration values for direct vapor partitioning measurements.

Before presenting the different approaches using empty or prepared vials, a discussion is included regarding the study of the preservation of soils by freezing. As noted, this was studied using empty VOA vials. Some of the empty VOA vial approaches that follow in Sec. A.8.2 use freezing as a preservative.

A.8.1 Overview of Empty Vial Technique

Hewitt (Refs. 2, 4, 7, 10) and Ricker (Refs. 19, 20, 21) independently developed "empty vial" techniques. Using a coring tool (Hewitt's cut-off syringe or Ricker's commercially available syringe and 5- to 13-g sample) a 5-g aliquot of undisturbed soil is transferred to a tared empty VOA vial and capped with a PTFE-lined septa (PTFE of 0.25 mm thickness). The two "empty vial" techniques were evaluated using methanol extraction (Method 5035) measurements.

The sealed vial with the soil aliquot is maintained either frozen ($< -7^\circ\text{C}$), or at $4 \pm 2^\circ\text{C}$ until laboratory receipt and analysis. Multiple VOA vials can be collected, as necessary based on the sample preparation technique to be used. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

Upon laboratory receipt of VOA vials maintained at $4 \pm 2^\circ\text{C}$ (within 48 hours of sample collection), one "empty VOA vial" is selected for methanol extraction and the methanol reagent is added through the septum using a glass syringe equipped with a 23-gage Luer Lock needle. The methanol is mixed with the soil and any pressure can be relieved by cracking the VOA vial's cap once. The methanol extract, stored at $4 \pm 2^\circ\text{C}$ or less, has a shelf life of up to two weeks. Upon laboratory receipt of frozen VOC samples, a vial may be thawed and methanol added through the septa as described above.

To determine VOCs by vapor partitioning, "empty VOA vials" should have 10 mLs of reagent water added, either through the septa liner by a laboratory's automated sampler at the time of analysis, or be present in the vial prior to sample collection (see Sec. A.8.3) when Method 5035 is used. For VOC samples maintained at $4 \pm 2^\circ\text{C}$ this must be done within 48 hours of sample collection. Experimental work by En Novative Technologies, Inc., and Hewitt (Refs. 44, 45) indicates that VOCs are slowly lost through the pierced septa after reagent water is manually added to an

"empty VOA vial," prior to Method 5035 purge and trap measurements. To avoid any clogging of the needle of an automated purge-and-trap system, reagent water or the sodium bisulfate solution can be present in the VOA vial (Sec. A.8.3) prior to sample collection, thereby, allowing the soil/solid to be dispersed prior to the purge-and-trap analysis.

Ricker and Hewitt in their experimental work demonstrated that the empty VOA vial, with a suitable PTFE-lined septa cap, has integrity for several days. Significant VOC losses do not occur at $4 \pm 2^\circ\text{C}$ through the septum of the sealed VOA vial. A 48-hour holding time for soils, at $4 \pm 2^\circ\text{C}$ storage of samples, has been found effective with the "empty VOA vial" for most target VOCs studied, except for aromatic compounds in biologically active, aerated garden soils (Refs. 2, 20). Hewitt studied freezing of soils ($< -7^\circ\text{C}$) as a preservative for soils, in conjunction with the "empty VOA vial" technique and found it effective for all target VOCs studied, including aromatic compounds, so long as freezing starts at the time of collection.

When soils are maintained at $4 \pm 2^\circ\text{C}$ for 48 hours until freezing starts, the same condition or stability is found for the VOCs except for benzene in biologically active soil. Use of freezing at the time of lab receipt of empty VOA vials can therefore simplify sample handling of soil materials. ASTM D 4547-98 (Ref. 15) and Method 5035 briefly mention freezing, but do not endorse it because data were not available at the time of their publication to support preservation by freezing. With this approach, chemical preservatives are not needed. VOA vials, maintained at $< -7^\circ\text{C}$, need only be thawed on the day of analysis, whether it be by vapor partitioning or by methanol extraction.

A.8.2 Preservation Approaches Using Empty VOA Vials

This section provides five examples of approaches to sample preparation using empty VOA vials -- no preservatives or solutions are added to the vials.

A.8.2.1 Preservation by freezing ($< -7^\circ\text{C}$)

Upon collection, the soil is added to replicate empty vials and frozen at $< -7^\circ\text{C}$ until thawed for analysis. The design of newer vials makes it possible to freeze the contents in an upright position, however, it may be advisable to place the vials on their side during the freezing process to prevent breakage. Freezing has been found effective to preserve both aromatic and chlorinated hydrocarbon VOCs in soil for two weeks at all VOC concentrations studied. (Refs. 2,4,11) Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

The on- or off-site support laboratory thaws a VOA vial when needed and either adds 5 or 10 mLs of methanol through the PTFE-lined septum using a 23-gage Luer lock syringe for methanol extraction and preservation. (Refs. 4,21,22) Addition of 5-10 mLs of water to the vial through the septum should not be performed, since this technique will create a punctured septum capable of producing VOC losses prior to purge-and-trap analysis.

This technique is unpopular for vapor partitioning because a prepared VOA vial with reagent water fits the operations of Methods 5021/5035 better than the empty VOA vial.

This technique can be undesirable when soil samples are transported to a support laboratory because dry ice, gel packs or salt-ice mixtures can be required to

maintain sub-zero temperature conditions during shipment. This technique has merit when freezers are available at a field site or on a sampling vessel.

A.8.2.2 Refrigerate VOA vials at $4 \pm 2^\circ\text{C}$ for 48 hours or less, then preserve by freezing at $< -7^\circ\text{C}$ upon laboratory receipt

Upon laboratory receipt, replicate soil VOA vials are frozen ($< -7^\circ\text{C}$) then thawed as needed for preparation by methanol extraction, or if possible by vapor partitioning. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. The 48-hour time period prior to freezing is practical and can be supported by the studies:

1. The chlorinated hydrocarbon volatiles that were studied have been found to be stable for two weeks at 4°C , with dichloroethene isomers not being as stable as other chlorinated compounds studied. (Refs. 1,2,4,7,11,16)
2. For spiked (at 5 ppm) typical soils, aromatic hydrocarbons demonstrate major losses at room temperature (22°C) after 5 days of storage. (Refs. 1,2,4) When these soil types are stored at 4°C , major losses occur between 10 and 14 days for aromatic hydrocarbons (e.g., benzene) spiked at 5 ppm. (Refs. 1,2,4) When these soil types are spiked at 30-40 ppb with aromatic hydrocarbons, major losses for benzene and toluene occur at 3-5 days of storage. (Refs. 2,4)
3. Aromatic hydrocarbons (such as benzene or toluene) when spiked into biologically active soil (aerated garden soil or fertilized soil) and stored at 4°C demonstrate losses of 20-30% within 48 hours. (Refs. 2,16,17,19,20). Limited disruption sampling techniques in conjunction with a maximum holding time of 48 hours can minimize this loss, but not eliminate it. Soils containing manure exhibited a major loss of aromatic hydrocarbons within one day while soil sterilization eliminated this loss. (Ref. 16)
4. Observed losses of aromatic or dichloroethene volatile compounds in soil, stored at 4°C , cease when soil is frozen at $< -7^\circ\text{C}$. (Refs. 2,4).

A.8.2.3 Refrigerate VOA vials at $4 \pm 2^\circ\text{C}$ for 48 hours or less, then preserve with methanol upon laboratory receipt

Upon laboratory receipt, the volume of methanol necessary for methanol extraction sample preparation is added to one of the replicate VOA vials through the PTFE-lined septum cap, using a 23-gage needle on a Luer lock syringe. Methanol will preserve VOCs in soil for 2 weeks if stored at $4 \pm 2^\circ\text{C}$. See Sec. A.8.2.2 above for discussion on initial ≤ 48 -hour transport at $4 \pm 2^\circ\text{C}$. Certain PTFE-lined septa caps were found to be effective seals for 10 days prior to the addition of methanol. (Refs. 19,20,21)

When methanol is added through the septum cap to a soil aliquot core in an empty VOA vial, the mixture is swirled to provide contact with the soil and methanol, to wet the head space, and dissolve gaseous and sorbed VOC compounds into the methanol. At this point, there can be a pressure build-up within the vial that can be removed by cracking the VOA vial cap and immediately resealing it. (Ref. 4) There is believed not to be significant VOC loss so long as the methanol remains in contact with

the soil material. The methanol extraction efficiency can be improved by sonicating and heating the mixture at 40°C for 30 minutes followed by centrifuging and transferring the supernatant to a disposable, screw-top glass centrifuge tube. (Ref. 33)

A.8.2.4 Refrigerate VOA vials at $4 \pm 2^\circ\text{C}$ for 48 hours or less and complete VOC analysis (Method 5021/5035) within 48 hours

VOC sample preparation by vapor partitioning is completed within 48 hours from sample collection. See Secs. A.8.2.2 and A.8.2.3 above for further details.

A.8.2.5 Refrigerate/freeze coring tool used as transport device for 48 hours or less (Refs. 15,26)

Each replicate soil aliquot is collected by a suitable coring device, (e.g., EnCore™) that is used as a transport device to the laboratory. Upon laboratory receipt, soil aliquots from each replicate transport device are extruded into individual empty or prepared tared VOA vials as noted in Secs. A.8.2.2 to A.8.2.4. Upon cap closure, the vial is weighed again and the wet sample weight is determined by difference.

For spiked soils characteristic of a waste site, some VOC losses were observed in 2 days for soils stored at $4 \pm 2^\circ\text{C}$ and losses continued further at a 5-day and 12-day storage time period. Losses during the first 2 days for aromatics and dichloroethene, were equivalent to the empty vial techniques as noted in Sec. A.8.2.2. (Ref. 4) Also, sampling of TCE contaminated soil showed reasonable agreement between the EnCore™ and cut-off syringe/empty vial techniques. (Ref. 4) Significant losses after 2 days at 4°C have been observed for the EnCore™ for biologically active soils. (Refs.16,24).

The EnCore™ sampler has been systematically evaluated for three sandy soil types (at high VOC concentrations (5 -10 ppm) and at low VOC concentrations (100 ppb). (Refs. 22,23,24,25). The EnCore™ was effective as a 2-day transport device when stored at $4 \pm 2^\circ\text{C}$, for the above studies, and storage could be extended from 1 week to 12 days further under freezing conditions ($< -7^\circ\text{C}$) during the low VOC concentration study. (Ref. 25) The EnCore™ was ineffective for one soil type using high concentration spikes, because the soil was non-cohesive (dry clumped sand) - any coring device could be ineffective. (Refs. 15,22) The three soils exhibited little biodegradation of aromatic hydrocarbons discussed above.

For the original EnCore™ of stainless steel construction, it was found to be the only sampling/storage device that was as effective as the original single vial technique (Dynatech vial of January 1995 draft Method 5035). (Ref. 16)

A.8.3 Preservation Approaches Using Prepared VOA Vials

This section provides four examples of preservation approaches using prepared VOA vials. During sample collection, a coring tool is used to extrude the collected sample into a VOA vial containing methanol. Co-located soil cores are extruded into replicate VOA vials containing reagent water, or reagent water acidified with 1 g NaHSO₄ per 5 mLs water.

Coordination between field and laboratory personnel is required so specific vials and reagents are consistent with laboratory instrumentation and reagents. Vials with reagents, and any magnetic stirring bars (e.g., for Method 5035) need be tared prior to field use. If prepared VOA vials contain methanol or water they must be tared with the septum caps and the added reagent. Once

methanol or water reagent is added, a meniscus level of the liquid in the VOA vial can be marked. This allows field personnel to note any apparent liquid loss (especially methanol) during shipment to the field. If field personnel are concerned with reagent weight loss during shipment to the field and return, individual vials can be periodically weighed after initial tare or after addition of cored soil aliquot.

A.8.3.1 Collection with reagent water, preservation by freezing ($< -7^{\circ}\text{C}$) and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs water (Method 5035), turn vial on its side and freeze contents. It may be problematical to freeze 10 mLs of water in the 22 ml vial used for Method 5021. Maintain at $< -7^{\circ}\text{C}$ until thawed for analysis. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing. Few published data exist to validate this preservation technique, but its effectiveness is inferred from Sec. A.8.2.1, and should be demonstrated by appropriate performance data results. (Ref. 28)

A.8.3.2 Collection with reagent water, preservation by refrigeration at $4 \pm 2^{\circ}\text{C}$ for 48 hours or less and immediate laboratory analysis or freezing storage at $< -7^{\circ}\text{C}$ for subsequent vapor partitioning

Sample is collected as in Sec. A.8.3.1 but transported to the laboratory within 48 hours at $4 \pm 2^{\circ}\text{C}$ for:

1. Immediate analysis by vapor partitioning within 48 hours of sample collection.
2. Freezing at $< -7^{\circ}\text{C}$ upon laboratory receipt for vapor partitioning analysis within 2 weeks from sample collection. Sample vials should not be frozen below -20°C due to potential problems with vial seals and the loss of constituents upon sample thawing.

One investigator has found that a spiked hazardous waste site soil provided the same results one week after freezing with water as the initial spiked soil results. (Ref 28). Another investigator used headspace techniques with soil added to 10 mLs of reagent water to develop justification for certain variables discussed in Sec. A.8.3.2 for the initial 48-hour holding time. (Refs. 6,12) Sec. A.8.2.2 should be consulted for biodegradation effects for aromatic hydrocarbons. This technique allows the laboratory to observe the dispersion of soils in water and take any corrective action prior to purge-and-trap analysis. This technique is also most consistent with automated purge-and-trap samplers where stirring occurs prior to the purge cycle.

A.8.3.3 Collection with 5 mLs of water and 1 g of NaHSO_4 and analysis by vapor partitioning

Extrude collected soil from a coring device into a VOA vial containing 5 mLs of reagent water and 1 g of NaHSO_4 for vapor partitioning by Method 5035. For a spiked soil, NaHSO_4 was found to preserve the aromatic hydrocarbons at room temperature for more than 2 weeks. (Refs.1,11) The same soil showed major losses of aromatic hydrocarbons (5-10 ppm) when stored at room temperature for 5 days or at $4 \pm 2^{\circ}\text{C}$ after 10 days when no NaHSO_4 was present. (Ref. 1) The studied chlorinated hydrocarbons demonstrated insignificant losses during these storage conditions. The

use of NaHSO_4 with sample acidification to pH 2 or less eliminates the biodegradation of the important aromatic hydrocarbon volatile compounds.

1 g of NaHSO_4 will acidify 5 g of soil with an alkaline content (as CaCO_3) of 5%. It is insufficient to neutralize a soil with an alkaline content of 10%. This technique has been found to be somewhat problematic since publication of Method 5035. Carbonaceous soils cause effervescence of the acidic soil slurry with loss of volatiles and even cause failure of the septa VOA vial cap or even the VOA vial itself. Upon acidification, certain soils exhibit a false positive acetone artifact of 100-200 ppb, or more. (Refs. 4,37) The NaHSO_4 corrosive vapors may cause increased purge-and-trap maintenance by laboratories due to creation of active sites on the trapping material. A very few target compounds such as styrene, vinyl chloride, and 2-chloroethylvinyl ether react under acidic conditions and are not detected. Note that the sodium chloride matrix modifying reagent of Method 5021 was found to be as effective as NaHSO_4 for inhibiting biodegradation of aromatic hydrocarbons in soil and may be more advantageous to use with calcareous soils, since the inhibitory agent is not dependent on the concentration of hydrogen ion present. (Ref. 1)

A.8.3.4 Collection and preservation with methanol at $4 \pm 2^\circ \text{C}$

Extrude collected soil from a coring device into a VOA vial containing 5 -10 mLs of methanol. Larger volumes of methanol may be used if compositing of soils is required. Methanol preservation is effective for 2 weeks if stored at $4 \pm 2^\circ \text{C}$. Also, one investigator has found methanol preservation of a sand spiked with gasoline to be effective when traditional techniques were ineffective. (Ref. 36)

1. An aqueous sample holding time period can be extended to fourteen days with the use of chemical preservatives such as sodium bisulfate or hydrochloric acid, however, since reactive compounds such as 2-chloroethylvinyl ether are unstable at low pHs, if these types of analytes are to be determined, the collection of a second set of samples without acid preservatives is necessary. Aqueous samples containing methyl *tert*-butyl ether and other fuel oxygenate ethers should not be acidified if high temperature sample preparative methods (Methods 5021, 5030, 5032) are used. (Refs. 49,50) (Sec. A.3.0)
2. The solid material to be characterized should be sampled with limited disruption (e.g., by a coring device for cohesive soils) and single transfer to an air tight VOA vial (PTFE-lined septa cap) that will be used for storage and preparation for VOC analysis. (Sec. A.7.1)
3. Data have been published or presented to validate different storage devices, procedures, preservative reagents and techniques for the VOC analysis of aqueous and solid samples. A wide range of recovery results have been observed. Acceptable devices, procedures, preservatives, and techniques should provide an average recovery of greater than 80% for important volatile contaminants such as benzene, dichloro- and trichloroethanes/ethenes. A recovery of 80% may be difficult for gaseous VOC contaminants such as vinyl chloride and chloroethane; however, the acceptability of a procedure should not be solely based on the less volatile VOCs such as chlorobenzene, xylenes, and trimethyl benzene. (Secs. A.2.0, A.6.0, A.7.0 and A.8.0)
4. VOCs in solids can be successfully sampled using coring tools (usually 5-g aliquots but can be 2 to 25 g) if the material is cohesive. Sampling procedures are not available to prevent VOC loss during sampling of non-cohesive soil material (dry sand, gravel, liquid sediment) or cemented material. (Secs. A.4.0 and A.7.0)
5. The following two techniques have been found accurate (minimal VOC loss) for preparation of soils for VOC analysis; however, they are not without problems:
 - a. Soil is added to empty VOA vials at time of collection and is frozen at $< -7^{\circ}\text{C}$ until thawed for analysis. Validation data have not been provided yet, but it is believed that a prepared VOA vial with reagent water only is also acceptable for low concentration VOC in soil (< 200 ppb) if frozen at $< -7^{\circ}\text{C}$ at time of collection.
 - b. Soil is added to a prepared VOA vial, with methanol reagent, at time of collection and stored at $4 \pm 2^{\circ}\text{C}$ until time of analysis. This is applicable only to VOC in soil concentrations greater than 50 ppb. (Sec. A.6.2) (See comments below regarding use of methanol.)
6. The following techniques have been found to be the most practical, currently available alternatives for preparation of soil for VOC analysis. Validation data are not available to fully support their use for all types of soil or to fully differentiate them in accuracy relative to each other. The techniques rely on transport of sealed VOA vials or coring tools, at $4 \pm 2^{\circ}\text{C}$, to a support laboratory within 48

hours where they are preserved/stored appropriately or immediately tested for VOCs. As more validation data and experience occur with time, their relative worth will become more apparent. The techniques listed below are superior to the traditional procedures of ten years ago.

- a. Soil is added to tared replicate "empty VOA vials" at time of collection, preserved, refrigerated at $4 \pm 2^\circ\text{C}$ until laboratory receipt within 48 hours, and then preserved by freezing ($< -7^\circ\text{C}$). Individual vials are thawed prior to sample preparation within 14 days of collection. A thawed vial must be processed within 24 hours by either screening using methanol extraction or analysis by vapor partitioning. At time of laboratory receipt, laboratories have the option of immediately testing a soil by vapor partitioning where the required reagent water is added through the PTFE-lined septa cap using the automated instrument sampling devices after weighing an "empty VOA vial" and obtaining wet sample weight by difference. In addition at time of laboratory receipt, laboratories have the option of immediately preparing a soil for methanol extraction by weighing an "empty VOA vial," obtaining the wet sample weight by difference, then adding methanol reagent through the PTFE-lined septa cap using a 23-gage needle on a Luer lock syringe. The sample-methanol mixture is shaken for 15 seconds to wet the vial's head space. The vial cap is opened once to vent pressure and then closed for the extraction process. (Sec. A.8.0)
- b. For carbonate-containing soils (or soils suspected as such), ASTM D4547-98 (Ref. 15) provides for adding 2 to 5 g of soil (using coring tool) to tared, replicate prepared VOA vial containing 5 mLs of reagent water. Prepared VOA vials are maintained at $4 \pm 2^\circ\text{C}$ until laboratory receipt within 48 hours, and immediately tested for VOCs by vapor partitioning. This approach offers the advantage of mixing and dispersing the soil into the water and to observe any problematic samples prior to vapor partitioning analysis. Alternatively, the reagent water prepared VOA vials may be preserved by freezing ($< -7^\circ\text{C}$) by placing vials in horizontal position. This technique is an alternative or fall-back from the prepared VOA vial with acidified reagent water; however, little or no data are available to validate its use. (Sec. A.5.3)
- c. Soil is collected in replicate "Coring Tool Used as Transport Device" (e.g., the EnCore™ sampler), maintained at $4 \pm 2^\circ\text{C}$ until laboratory receipt within 48 hours, then extruded into individual "Empty VOA Vials" for preservation by freezing ($< -7^\circ\text{C}$) or into prepared VOA vials for immediate analysis by vapor partitioning or for sample preparation by methanol extraction. (Sec. A.7.2)
- d. For known non-carbonate soils, a coring tool soil aliquot for BTEX type VOC analysis is added to a tared prepared VOA vial containing 5 mLs reagent water acidified with 1g NaHSO_4 . The prepared VOA vial is maintained at $4 \pm 2^\circ\text{C}$ for BTEX testing by vapor partitioning within 14 days of sample collection. Acidified reagent water has been problematic when applied to a wide range of soil types for a large analyte list; however, it is effective for the volatile BTEX compounds in known non-carbonate soils. It is a specialized, preservation technique that minimizes aromatic VOC losses from biodegradation at $4 \pm 2^\circ\text{C}$.

Acetone artifacts are sometimes observed in soil samples preserved with NaHSO₄.

7. Use of a prepared VOA vial with acidified (NaHSO₄) reagent water is not recommended as a primary preservation technique for all soil types and a broad VOC analyte list. This technique is applicable to volatile aromatic hydrocarbons in soils known not to contain carbonates as discussed above.
8. A longer holding time may be appropriate if it can be demonstrated that the reported VOC concentrations are not adversely affected from storage and analyses performed outside the recommended holding times.

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APPENDIX B

FSP/QAPP

ELECTRONIC DATA DELIVERABLE SPECIFICATIONS

MWH ELECTRONIC DATA DELIVERABLE REQUIREMENTS

(rev. March 2011)

Field Name	Format	Description	Constraints	Comments
AFIID	C5 (Valid Value List)	USAF Installation Code	Required	
LABSAMPID	C20	Lab Sample Identifier	Required	
LOCID	C15	Location Name	Required	
MATRIX	C2 (Valid Value List)	Sampling Matrix	Required	
SBD	N8,2	Sample Beginning Depth	Required	
SED	N8,2	Sample Ending Depth	Required	
LOGDATE	D11 (DD-MMM-YYYY)	Sample Date	Required	
LOGTIME	C4 (HHMM)	Sample Time	Required	
LABCODE	C4 (Valid Value List)	USAF Lab Identifier	Required	
SACODE	C2 (Valid Value List)	Sample Type	Required	
SAMPNO	N2,0	Sample Number	Required	
ANMCODE	C7 (Valid Value List)	Analytical Method Code	Required	
EXMCODE	C7 (Valid Value List)	Extraction Method Code	Required	
EXTDATE	D11 (DD-MMM-YYYY)	Extraction Date	Conditional	
EXTTIME	C4 (HHMM)	Extraction Time	Conditional	
ANADATE	D11 (DD-MMM-YYYY)	Analysis Date	Required	
ANATIME	C4 (HHMM)	Analysis Time	Required	
LABLOTCTL	C10	Laboratory Preparation Batch ID	Required	
PARLABEL	C12 (Valid Value List)	Parameter Label	Required	
PARVAL	N16,6	Measured Concentration	Required	
UNITS	C10 (Valid Value List)	Units of Measure	Required	
PARVQ	C2 (Valid Value List)	Parameter Value Qualifier	Required	
BASIS	C1 (Valid Value List)	Wet or Dry Basis (for soil data)	Required	
DILUTION	N16,6	Dilution Factor	Required	
LOGCODE	C4 (Valid Value List)	Logging Company Code	Required	Should equal "MWSL"
SMCODE	C2 (Valid Value List)	Sampling Method Code	Required	
FLDSAMPID	C30	Field Sample ID	Required	
COCID	C12	Chain of Custody ID	Optional	
COOLER	C10	Field Cooler ID	Optional	
ABLOT	C8 (DDMMYYNN)	Ambient Blank Lot ID	Optional	
EBLOT	C8 (DDMMYYNN)	Equipment Blank Lot ID	Optional	
TBLOT	C8 (DDMMYYNN)	Trip Blank Lot ID	Optional	
PARUN	N12,4	Uncertainty	Conditional	
PRECISION	N1	Primary Value Precision	Required	
EXPECTED	N16,6	Expected Value (for spiked samples)	Conditional	
EVPREC	N1	Expected Value Precision	Conditional	
MDL	N16,6	Method Detection Limit	Conditional	
RL	N16,6	Reporting Limit	Conditional	
LCHMETH	C7 (Valid Value List)	Leachate Method	Required	
RUN_NUMBER	N2	Run Number	Required	
LCHDATE	D11 (DD-MMM-YYYY)	Leachate Date	Conditional	
LCHTIME	C4 (HHMM)	Leachate Time	Conditional	
LCHLOT	C10	Leachate Lot	Conditional	
ANALOT	C10	Analytical Lot	Required	
PRCCODE	C3 (Valid Value List)	Analyte Type	Required	
CALREFID	C10	Calibration Reference	Optional	
VQ_1C	C2 (Valid Value List)	PARVQ of first Column	Optional	
VAL_1C	N16,6	Result of first Column	Optional	
FCVALPREC	N1	Precision of first Column	Optional	
VQ_CONFIRM	C2 (Valid Value List)	PARVQ of Confirmation Column	Optional	
VAL_CONFIRM	N16,6	Result of Confirmation Column	Optional	
CNFVALPREC	N1	Precision of Confirmation Column	Optional	
LAB_DQT	C5 (Valid Value List)	Type of Data Qualifier System	Optional	To be provided by MWH project chemist
LAB_QC_FLAG	C6 (Valid Value List)	Laboratory Flags	Conditional	Previously referred to as QAPP_FLAG
REC_DATE	D11 (DD-MMM-YYYY)	Date Sample Received in Lab	Required	
COMPNAME	C50	Compound Name	Required	Not an ERPIMS field

MWH ELECTRONIC DATA DELIVERABLE REQUIREMENTS (rev. March 2011)

Field Name	Format	Description	Constraints	Comments
CASNUMBER	C10	Chemical Abstract Service No.	Optional	Not an ERPIMS field
SPIKE_ADDED	N16,6	Concentration Spiked	Conditional	
PRIME_FLAG	C6	Validation Qualifiers	Leave Null	Previously referred to as EPA_FLAGS
LOWER_ACCURACY	N14,2	Minimum Precision Control Limit	Conditional	Previously referred to as LOW_LIMIT
UPPER_ACCURACY	N14,2	Maximum Precision Control Limit	Conditional	Previously referred to as HIGH_LIMIT
UPPER_RPD	N14,2	Maximum RPD control limit	Conditional	Previously referred to as RPD
PERCENT_RECOVERY	N14,2	Percent Recovery	Conditional	New field requirement

Notes:

An Excel file is preferred, however, a single delimited text file (.csv) is acceptable. If a csv file is used the delimiters may be commas with quote text qualifiers ("VAL1","VAL2") or tabs.

It is required that all fields be provided in the order listed above, a place-holder must be provided for any null entries.

The latest ERPIMS DLH (Data Loading Handbook) version must be used to obtain valid values.

The format column lists the data type ([N]umber, [C]haracter, [D]ate) followed by the number of allowed characters. For number data types the number of decimal places is indicated by the number following the comma. Additional format constraints are listed within parenthesis.

The PARVAL field is the actual concentration (not percent recovery) unless the PRCCODE = STD (surrogate results).

The LOGDATE and LOGTIME fields are required, for LABQC samples use the earlier of the EXMDATE/TIME or ANMDATE/TIME.

Conditional Constraints:

FLDSAMPID should repeat the LABSAMPID for LABQC samples.

EXTDATE, EXTTIME are required unless the EXMCODE = "NONE"

LCHDATE, LCHTIME and LCHLOT are required unless the LCHMETH = "NONE"

PERCENT RECOVERY is required whenever the SACODE = MS,SD,BS, or BD. It should not be populated when the PRCCODE = STD (surrogates).

PARUN is required only when PRCCODE = RN (Radionuclides); it should not be populated in any other instance.

MDL and RL are required for all results unless the PRCCODE = MI,PM,BAC or STD. Do not populate MDL and RL for TICs (PARVQ = TI). When QSM is used RL = LOQ and MDL = LOD.

EXPECTED is required when SACODE <> 'N'. EXPECTED is required for records with a PRCCODE = STD regardless of SACODE. EXPECTED is reported in the same units as PARVAL.

EVPREC should be populated each time an EXPECTED value is required.

LAB_QC_FLAG is required when a flag is needed.

SPIKE ADDED, LOWER AND UPPER ACCURACY are required whenever the SACODE = MS,SD,BS, or BD. It is also required whenever the PRCCODE = STD.

UPPER_RPD is required for all records with a SACODE of LR,SD,BD,FR,or FD unless the PRCCODE = STD.

APPENDIX C

FSP/QAPP

DATA VALIDATION REPORT TEMPLATES

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Monsanto, P4 Production LLC

Report Date: September 29, 2010

Matrix: Water

Parameters: Volatiles by GC/MS SW-846 Method 8260B

Validation Level: Stage 4

Laboratory: Laboratories, Inc.

Sample Delivery Group (SDG): 1231236

Sample Identification	Collection Date	Laboratory Sample Identification
FRANK-GW169	08/17/10	1231236-01
FRANK-GW171	08/17/10	1231236-02
FRANK-GW167	08/17/10	1231236-03
FRANK-EQ400	08/17/10	1231236-04
SMITH-GW166	08/18/10	1231236-05
SMITH-GW400	08/18/10	1231236-06
SMITH-GW164	08/18/10	1231236-07
SMITH-GW162	08/19/10	1231236-08
SMITH-GW172	08/19/10	1231236-11
SMITH-GW161	08/19/10	1231236-12
SMITH-EQ400	08/19/10	1231236-13
SMITH-GW160	08/19/10	1231236-14
SMITH-GW159	08/19/10	1231236-15
SMITH-GW162-MS	08/19/10	1231236-09
SMITH-GW162-MSD	08/19/10	1231236-10

Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA SW 846 Method noted below:

- Method 8260B GC/MS: Volatile organic compounds (VOCs)

This review follows the specific guidance in the *Ballard Mine Shop Sampling and Analysis Plan* (SAP; MWH 2011) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

- | | |
|----|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| R | The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |

The following are not data qualifiers but are provided for the purpose of evaluating the laboratory's performance:

- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

- 1 Holding Time
- 2 Sample Preservation (including receipt temperature)
- 3 Sample Custody
- 4 Missing Deliverable
- 5 Instrument Performance Check
- 6 Initial Calibration
- 7 Initial Calibration Verification
- 8 Continuing Calibration Verification
- 9 Low-Level Calibration Check Sample
- 10 Calibration Blank
- 11 Laboratory or Preparation Blank
- 12 Surrogate spikes
- 13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
- 14 Laboratory Control Sample Precision
- 15 Laboratory Duplicate Precision
- 16 Matrix Spike or Matrix Spike Duplicate Recovery
- 17 Matrix Spike/Matrix Spike Duplicate Precision
- 18 Internal Standard
- 19 Field Replicate Precision
- 20 Equipment Rinsate Blank
- 21 Linear Range Exceeded
- 22 Other reason
- 23 Source Water Blank
- 24 Result is less than the MDC
- 25 Result is less than two times the error
- 26 Source Water Blank
- 27 Surrogate
- 28 Peak Resolution
- 29 Trip Blank

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections performed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements were met: 14 days for analysis (soil and water).

All samples were received intact at proper temperatures and with proper preservation (pH < 2 for water and sodium bisulfate and methanol for soil).

II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals. All ion abundance requirements were met.

III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 30.0 for calibration check compounds (CCCs) and 15 for all other compounds.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990.

Second-source initial calibration verification (ICV) percent differences with ± 20 of the expected value.

Average relative response factors (RRF) for all compounds were within method and validation criteria.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0% all compounds.

All of the continuing calibration RRFs was within method and validation criteria.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No VOCs were found in either the method blanks.

Samples FRANK-EQ400 and SMITH-EQ400 were identified as rinsates. No volatile contaminants were found in these blanks.

VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries were within the project-specific QC limits (as specified on Table 4-13 of Appendix A of the SAP).

VII. Laboratory Control Sample (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries were within the project-specified control limits (as specified on Table 4-13 of Appendix A of the SAP).

VIII. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within the project-specified control limits (as specified on Tables 4-8 and 4-13 of Appendix A of the SAP).

IX. Internal Standards

All internal standard areas and retention times were within the method limits as specified on Table 4-9 of Appendix A of the SAP (RT \pm 30 seconds from RT of the midpoint standard in the ICAL; Extracted Ion Current Profile (EICP) area within -50% to +100% of ICAL midpoint standard).

X. Field Replicates

Field replicate samples were collected in duplicate. All RPDs were less than or equal to 20 for water samples and 35 for soil samples.

XI. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

VOCs - Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

VOCs - Laboratory Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

VOCs - Field Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

Laboratory Data Consultants, Inc. Data Validation Report

Project/Site Name: Monsanto, P4 Production LLC

Report Date: September 29, 2010

Matrix: Water

Parameters: Semivolatiles by GC/MS SW-846 Method 8270C

Validation Level: Stage 4

Laboratory: Laboratories, Inc.

Sample Delivery Group (SDG): 1231236

Sample Identification	Collection Date	Laboratory Sample Identification
FRANK-GW169	08/17/10	1231236-01
FRANK-GW171	08/17/10	1231236-02
FRANK-GW167	08/17/10	1231236-03
FRANK-EQ400	08/17/10	1231236-04
SMITH-GW166	08/18/10	1231236-05
SMITH-GW400	08/18/10	1231236-06
SMITH-GW164	08/18/10	1231236-07
SMITH-GW162	08/19/10	1231236-08
SMITH-GW172	08/19/10	1231236-11
SMITH-GW161	08/19/10	1231236-12
SMITH-EQ400	08/19/10	1231236-13
SMITH-GW160	08/19/10	1231236-14
SMITH-GW159	08/19/10	1231236-15
SMITH-GW162-MS	08/19/10	1231236-09
SMITH-GW162-MSD	08/19/10	1231236-10

Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA SW 846 Method noted below:

- Method 8270C GC/MS: Semivolatiles organic compounds (SVOCs)

This review follows the specific guidance in the *Ballard Mine Shop Sampling and Analysis Plan* (MWH 2011) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

- | | |
|----|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| R | The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |

The following are not data qualifiers but are provided for the purpose of evaluating the laboratory's performance:

- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

- 1 Holding Time
- 2 Sample Preservation (including receipt temperature)
- 3 Sample Custody
- 4 Missing Deliverable
- 5 Instrument Performance Check
- 6 Initial Calibration
- 7 Initial Calibration Verification
- 8 Continuing Calibration Verification
- 9 Low-Level Calibration Check Sample
- 10 Calibration Blank
- 11 Laboratory or Preparation Blank
- 12 Surrogate spikes
- 13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
- 14 Laboratory Control Sample Precision
- 15 Laboratory Duplicate Precision
- 16 Matrix Spike or Matrix Spike Duplicate Recovery
- 17 Matrix Spike/Matrix Spike Duplicate Precision
- 18 Internal Standard
- 19 Field Replicate Precision
- 20 Equipment Rinsate Blank
- 21 Linear Range Exceeded
- 22 Other reason
- 23 Source Water Blank
- 24 Result is less than the MDC
- 25 Result is less than two times the error
- 26 Source Water Blank
- 27 Surrogate
- 28 Peak Resolution
- 29 Trip Blank

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections performed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements were met: 7 days for water extraction and 14 days for soil extraction, 40 days from extraction to analysis for both soil and water.

All samples were received intact at proper temperatures.

II. GC/MS Instrument Performance Check

Instrument performance was checked at 12 hour intervals. All ion abundance requirements were met.

III. Initial Calibration

Initial calibration was performed using required standard concentrations.

Percent relative standard deviations (%RSD) were less than or equal to 30.0 for calibration check compounds (CCCs) and 15 for all other compounds.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990.

Second-source initial calibration verification (ICV) percent differences with ± 20 of the expected value.

Average relative response factors (RRF) for all compounds were within method and validation criteria.

IV. Continuing Calibration

Continuing calibration was performed at the required frequencies.

Percent differences between the initial calibration RRF and the continuing calibration RRF were within the method criteria of less than or equal to 20.0 for all compounds.

All of the continuing calibration relative response factors (RRF) were within method and validation criteria.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No SVOCs were found in the method blanks.

Samples FRANK-EQ400 and SMITH-EQ400 were identified as rinsates. No polychlorinated biphenyl contaminants were found in these blanks.

VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within the project-specified control limits (as specified on Table 4-14 of Appendix A of the SAP).

VII. Laboratory Control Sample (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within the project-specified control limits (as specified on Table 4-14 of Appendix A of the SAP).

VIII. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within the project-specified control limits (as specified on Tables 4-9 and 4-14 of Appendix A of the SAP).

IX. Internal Standards

All internal standard areas and retention times were within the method limits as specified on Table 4-9 of Appendix A of the SAP ($RT \pm 30$ seconds from RT of the midpoint standard in the ICAL; Extracted Ion Current Profile (EICP) area within -50% to +100% of ICAL midpoint standard).

X. Field Replicates

Field replicate samples were collected in duplicate. All RPDs were less than or equal to 20 for water samples and 35 for soil samples.

XI. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

SVOCs - Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

SVOCs - Laboratory Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

SVOCs - Field Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

Laboratory Data Consultants, Inc.
Data Validation Report

Project/Site Name: Monsanto, P4 Production LLC

Report Date: September 29, 2010

Matrix: Water

Parameters: Polychlorinated Biphenyls by GC SW-846 Method 8082

Validation Level: Stage 4

Laboratory: Laboratories, Inc.

Sample Delivery Group (SDG): 1231236

Sample Identification	Collection Date	Laboratory Sample Identification
FRANK-GW169	08/17/10	1231236-01
FRANK-GW171	08/17/10	1231236-02
FRANK-GW167	08/17/10	1231236-03
FRANK-EQ400	08/17/10	1231236-04
SMITH-GW166	08/18/10	1231236-05
SMITH-GW400	08/18/10	1231236-06
SMITH-GW164	08/18/10	1231236-07
SMITH-GW162	08/19/10	1231236-08
SMITH-GW172	08/19/10	1231236-11
SMITH-GW161	08/19/10	1231236-12
SMITH-EQ400	08/19/10	1231236-13
SMITH-GW160	08/19/10	1231236-14
SMITH-GW159	08/19/10	1231236-15
SMITH-GW162-MS	08/19/10	1231236-09
SMITH-GW162-MSD	08/19/10	1231236-10

Introduction

This data review covers 15 water samples listed on the cover sheet including dilutions and reanalysis as applicable. The analysis was performed per the EPA SW 846 Method noted below:

- Method 8082 GC: Polychlorinated biphenyls (PCBs).

This review follows the specific guidance in the *Ballard Mine Shop Sampling and Analysis Plan* (MWH 2011) using the intent of the USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review (June 2008) as applicable to the method stated above.

A qualification summary table is provided at the end of this report if data has been qualified. Flags are classified as P (protocol) or A (advisory) to indicate whether the flag is due to a laboratory deviation from a specified protocol or is of technical advisory nature.

Raw data were reviewed for a minimum of 10% of the Sample Delivery Groups (SDGs) or laboratory data package deliverables associated with this sampling event as specified in the QAPP Addendum. This package includes raw data review.

The following are definitions of the data qualifiers:

- | | |
|----|---|
| U | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| J | The result is an estimated quantity. The associated numerical value is the approximated concentration of the analyte in the sample. |
| J+ | The result is an estimated quantity, but the result may be biased high. |
| J- | The result is an estimated quantity, but the result may be biased low. |
| R | The result is unusable. The sample result is rejected due to serious deficiencies in meeting quality control criteria. The analyte may or may not be present in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |

The following are not data qualifiers but are provided for the purpose of evaluating the laboratory's performance:

- A Indicates the finding is based upon technical validation criteria.
- P Indicates the finding is related to a protocol/contractual deviation.

The following "Reason Codes" will be applied as applicable to the validated data:

- 1 Holding Time
- 2 Sample Preservation (including receipt temperature)
- 3 Sample Custody
- 4 Missing Deliverable
- 5 Instrument Performance Check
- 6 Initial Calibration
- 7 Initial Calibration Verification
- 8 Continuing Calibration Verification
- 9 Low-Level Calibration Check Sample
- 10 Calibration Blank
- 11 Laboratory or Preparation Blank
- 12 Surrogate spikes
- 13 Laboratory Control Sample or Laboratory Control Sample Duplicate Recovery
- 14 Laboratory Control Sample Precision
- 15 Laboratory Duplicate Precision
- 16 Matrix Spike or Matrix Spike Duplicate Recovery
- 17 Matrix Spike/Matrix Spike Duplicate Precision
- 18 Internal Standard
- 19 Field Replicate Precision
- 20 Equipment Rinsate Blank
- 21 Linear Range Exceeded
- 22 Other reason
- 23 Source Water Blank
- 24 Result is less than the MDC
- 25 Result is less than two times the error
- 26 Source Water Blank
- 27 Surrogate
- 28 Peak Resolution
- 29 Trip Blank

I(a). Deliverables and Chain-of-Custody Documentation

All deliverables were present and complete including the Case Narrative with full explanation of corrective actions and all package deliverables defined in the project SAP.

The chain-of-custodies were complete for sample identification, matrix, methods, preservation, dates and times of collection, dates and times of relinquishment and receipt. Any corrections performed properly (i.e., crossed-out with a single line; correction visible, neat, and clear; and with initials of individual making correction).

I(b). Preservation and Holding Times

All technical holding time requirements were met: 7 days for water extraction and 14 days for soil extraction, 40 days from extraction to analysis for both soil and water.

All samples were received intact at proper temperatures.

II. GC/ECD Instrument Performance Check

Instrument performance was acceptable unless noted otherwise under initial calibration and continuing calibration sections.

III. Initial Calibration

Initial calibration of multicomponent compounds was performed for the primary (quantitation) column as required by the method.

The percent relative standard deviations (%RSD) were less than or equal to 20.0 for all compounds.

In the case where the laboratory used a calibration curve to evaluate the compounds, all coefficients of determination (r^2) were greater than or equal to 0.990.

The percent differences of the second-source initial calibration verification (ICV) standard were less than or equal to 20.0 for all compounds.

IV. Continuing Calibration

Continuing calibration was performed at required frequencies.

The percent differences of calibration factors in continuing standard mixtures were within the 20.0 for each mixture.

V. Blanks

Method blanks were reviewed for each matrix as applicable. No PCBs were found in the preparation blanks.

Samples FRANK-EQ400 and SMITH-EQ400 were identified as rinsates. No

polychlorinated biphenyl contaminants were found in these blanks.

VI. Surrogate Spikes

Surrogates were added to all samples and blanks as required by the method. All surrogate recoveries (%R) were within project-specified control limits (as specified on Table 4-15 of Appendix A of the SAP).

VII. Laboratory Control Sample (LCS)

Laboratory control samples were reviewed for each matrix as applicable. Spike amounts were reviewed and concentrations are noted to be at or near the mid-point of the calibration. Percent recoveries (%R) were within the project-specified control limits (as specified on Table 4-15 of Appendix A of the SAP).

VIII. Spike Sample Analysis

Matrix spike (MS) and matrix spike duplicate (MSD) samples were reviewed for each matrix as applicable. Percent recoveries (%R) and relative percent differences (RPD) were within the project-specified limits (as specified on Tables 4-10 and 4-15 of Appendix A of the SAP).

IX. Target Compound Identification

The retention times (RTs) of both of the surrogates and reported target compounds are within the calculated RT windows on both columns. The surrogate TCX is within ± 0.05 minutes of the mean RT determined from the ICAL, and the surrogate DCB is within ± 0.10 minutes of the mean RT determined from the ICAL. The percent difference for the detected mean concentrations of the target compounds between the two GC columns is with ± 25.0 . Chromatographic patterns of samples containing one or more PCB are comparable to patterns of the standards.

X. Field Replicates

Field replicate samples were collected in duplicate. All RPDs were less than or equal to 20 for water samples and 35 for soil samples.

XI. Overall Assessment of Data

Data flags are summarized at the end of this report if data has been qualified.

PCBs - Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

PCBs - Laboratory Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

PCBs - Field Blank Data Qualification Summary - SDG 1231236

No Sample Data Qualified in this SDG

BALLARD SHOP SAP

APPENDIX B

HEALTH AND SAFETY PLAN ACTIVITY HAZARD ANALYSIS

Tasks	Hazards	Controls	PPE Required
Sampling at Operating Mine Sites (including sites undergoing reclamation)	<ul style="list-style-type: none"> Cuts and scrapes 	<ul style="list-style-type: none"> Follow procedures of mine operator. Report injuries to buddy or to person designated by mine operator for first aid if necessary. Come to work alert and ready—make sure that general awareness of surroundings is part of job planning and execution. Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground. 	<p>Minimum: hard-hat, safety glasses, boots, long pants, and cotton shirt; heavy work gloves for handling sharp objects.</p> <p>Additional PPE as specified by the mine operator.</p>
	<ul style="list-style-type: none"> Heat or cold stress 	<ul style="list-style-type: none"> Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0). 	
	<ul style="list-style-type: none"> Slips/trips/falls 	<ul style="list-style-type: none"> Maintain general awareness of surroundings. 	
	<ul style="list-style-type: none"> Being struck by heavy equipment or caught between equipment and a stationary object 	<ul style="list-style-type: none"> Receive site-specific hazard training. Be alert to the direction of traffic flow. Maintain eye contact with heavy equipment operators and give them the right-of-way. Never stand between operating vehicles and nearby stationary objects. Ask the mine operator where the blind spots for each piece of equipment are located— DO NOT STAND IN BLIND SPOTS. 	

Tasks	Hazards	Controls	PPE Required
Sampling at Operating Mine Sites (continued)	<ul style="list-style-type: none"> High wall collapse 	<ul style="list-style-type: none"> Receive site-specific hazard training. Perform work under escort of mine employee. Do not stand between high wall and heavy equipment—make sure you have an escape route. Know the mine emergency signals and evacuation procedures. 	
Sampling at Inactive Mine Sites	<ul style="list-style-type: none"> Cuts and scrapes Slips/trips/falls Dislodged rocks 	<ul style="list-style-type: none"> Report injuries to buddy for first aid if necessary. Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground. Do not walk at the edge of sharp drop-offs. Maintain special care on scree slopes or while working in other areas with unstable footing. Maintain general awareness of surroundings. Be aware of the possibility of abandoned underground mine portals. Avoid areas below people who may dislodge rocks while working or walking on slopes. Cry “ROCK” after dislodging a rock when other people are below. 	Minimum: hard-hat, boots, long pants, and cotton shirt; heavy work gloves for handling sharp objects.

Tasks	Hazards	Controls	PPE Required
Sampling at Inactive Mine Sites (continued)	<ul style="list-style-type: none"> Deteriorated roads 	<ul style="list-style-type: none"> Receive site-specific hazard training. Exercise care while traveling by vehicle. 	
	<ul style="list-style-type: none"> High wall collapse or rock-fall 	<ul style="list-style-type: none"> Receive site-specific hazard training. Know signs of instability. Carefully examine the surroundings to determine if entry is safe. Be aware of the most efficient evacuation route. Do not walk on top of high walls. Avoid working downslope of rock slides. 	
	<ul style="list-style-type: none"> Heat or cold stress 	<ul style="list-style-type: none"> Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0). 	
	<ul style="list-style-type: none"> Drinking water from streams, mine pits, mine ponds 	<ul style="list-style-type: none"> Water in mine pits is of unknown quality, and WILL NOT be used for drinking water. Water purification with iodine, filters, or boiling will not remove potentially toxic metals. 	

Tasks	Hazards	Controls	PPE Required
Soil boring drilling and soil sampling using Hollow-Stem Auger drilling equipment	<ul style="list-style-type: none"> • Possibility of exposure to petroleum hydrocarbons, chlorinated hydrocarbons, radionuclide's, PCBs, PAHs and metals 	<ul style="list-style-type: none"> • Breathing zone air monitoring using a PID 	Minimum: hard-hat, boots, long pants, and cotton shirt; heavy work gloves for handling sharp objects. Level D or C PPE with upgrading or downgrading pending observed site conditions and monitoring results for volatile organic compounds
	<ul style="list-style-type: none"> • Heavy lifting (augers and bags of bentonite) 	<ul style="list-style-type: none"> • When lifting, be sure to size up the load, get assistance when possible and follow proper lifting techniques. If possible use sling or strap while handling augers 	
	<ul style="list-style-type: none"> • Being struck by equipment/vehicles 	<ul style="list-style-type: none"> • Never approach the backhoe or excavator without making eye contact and being signaled to approach by the backhoe operator 	
	<ul style="list-style-type: none"> • Contact with overhead obstructions 	<ul style="list-style-type: none"> • Check for overhead obstructions prior to raising mast of rig or extending backhoe arm. Maintain a 20-foot clearance from overhead power lines. Check underground utility clearance with both on-site and off-site utility locators. 	
	<ul style="list-style-type: none"> • Slip/trip/fall 	<ul style="list-style-type: none"> • Watch where you step, be aware of uneven terrain. Keep footwear and work area free of mud and drilling fluids. Maintain 3 points of contact when mounting and dismounting drill rig or backhoe. 	

Tasks	Hazards	Controls	PPE Required
		<ul style="list-style-type: none"> Do not climb on the drill rig above 6 feet without the use of fall protection. All unattended bore holes must be covered or properly abandoned to ground surface. Watch your footing to prevent slips and avoid stepping between augers and drill steel to prevent injuries. 	
		<ul style="list-style-type: none"> Always keep fingers clothing and tools clear of auger guides. This should be done only when rotation and feet controls are in neutral. Do not place hands or fingers under the bottom of an auger section when hoisting the auger over the top of another section or any hard object. Whenever possible use tools to hoist handle augers. Never put fingers in bolt-holes to clean threads while augers are coupled. Do not attempt to remove cuttings from rotating augers. Always keep loose clothing away from augers and rotating parts. 	
		<ul style="list-style-type: none"> Catheads are dangerous. Always use a clean-dry rope. Never use a rope longer than necessary. Never leave the rope on the drum. The cathead should be replaced if a rope groove of greater than 1/8 inch forms. Many times in wet or icy conditions a cathead cannot be used. Always operate the drill from the control platform. The operator must never leave the control panel while the drill is in operations. 	

Tasks	Hazards	Controls	PPE Required
		<ul style="list-style-type: none"> Never spray pressure washer in the direction of any on site personnel. Avoid any contact with spray stream of water. 	
Travel in Remote Areas	<ul style="list-style-type: none"> General 	<ul style="list-style-type: none"> Always carry ten essentials for wilderness travel (see Table 2-3). 	Heavy work gloves for handling sharp objects.
	<ul style="list-style-type: none"> Slips/trips/falls 	<ul style="list-style-type: none"> Maintain general awareness of surroundings. 	
	<ul style="list-style-type: none"> Cuts and scrapes 	<ul style="list-style-type: none"> Report injuries to buddy for first aid. Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. Wear heavy work gloves when handling sharp objects, and point sharp objects toward the ground. 	
	<ul style="list-style-type: none"> Safe drinking water 	<ul style="list-style-type: none"> Contact National Forest officials in advance regarding any water quality advisories. Bring sufficient water. Assume that you will need one gallon of drinking water per person per day. 	
	<ul style="list-style-type: none"> Severe weather 	<ul style="list-style-type: none"> Bring proper rain gear and warm clothes. Listen to weather forecasts before entering remote areas. If severe weather is likely, postpone sampling. In case of lightning, avoid high ground and open areas. In the event of rain, monitor for 	

Tasks	Hazards	Controls	PPE Required
Travel in Remote Areas (continued)		hypothermia.	
		<ul style="list-style-type: none"> In the event of snow, monitor for frostbite and hypothermia. In the event of a blizzard that reduces visibility, stay put in an emergency shelter. Do not risk disorientation. 	
	<ul style="list-style-type: none"> Getting lost 	<ul style="list-style-type: none"> Provide the Program Manager or designee with itineraries, including travel routes and the expected date and time of return. Check in once per day, if possible, when in remote areas. Always check in with the Program Manager or designee before and after sampling. The Program Manager or designee will contact search and rescue if field personnel do not return or call in by the specified time. Bring emergency shelter. If lost, stay put. You are easier to find this way. 	
	<ul style="list-style-type: none"> Heat or cold stress 	<ul style="list-style-type: none"> Monitor for heat or cold stress as outlined in the Health and Safety Plan (see Section 7.0). 	
	<ul style="list-style-type: none"> Muscle strains Poisonous plants and animals 	<ul style="list-style-type: none"> Know your limits, and do not overextend yourself. Be able to recognize poisonous plants and animals and avoid them. If bitten by a snake or spider, apply cold compresses. Get to a hospital as quickly as 	

Tasks	Hazards	Controls	PPE Required
Travel in Remote Areas (continued)	<ul style="list-style-type: none"> Wildlife 	<p>possible.</p> <ul style="list-style-type: none"> Avoid, if possible, and leave the area. Make yourself look large by raising arms and shouting. Slowly back away, without turning your back to the animal. 	

Tasks	Hazards	Controls	PPE Required
General Work Practices	• First aid injuries	<ul style="list-style-type: none"> • Report injuries to buddy for first aid. • Seek additional medical attention, if necessary. • Notify the PSO. 	Minimum: hard-hat, safety glasses, boots, long pants, and cotton shirt.
	• Slips/trips/falls	<ul style="list-style-type: none"> • Practice good housekeeping, and remove or reduce slip/trip/fall hazards. • Maintain general awareness of surroundings. 	Additional: heavy work gloves and hearing protection, as necessary.
	• Cuts/scrapes	<ul style="list-style-type: none"> • Report injuries to buddy for first aid. • Come to work alert and ready—make sure that general awareness of site surroundings is part of job planning and execution. • Wear heavy work gloves when handling sharp objects and point sharp objects towards the ground. 	
	• Heat or cold stress	<ul style="list-style-type: none"> • Monitor for heat and cold stress as outlined in the Health and Safety Plan (see Section 7.0). 	
	• Muscle strain	<ul style="list-style-type: none"> • Alternate activities as needed to give muscles rest. 	
	• Slips/trips/falls	<ul style="list-style-type: none"> • Practice good housekeeping to remove or reduce slip/trip/fall hazards. 	
	• Hearing loss	<ul style="list-style-type: none"> • Use hearing protection when operating loud equipment. 	

Tasks	Hazards	Controls	PPE Required
General Work Practices (continued)	• Electrocution	• Use GFCI on portable power equipment.	
	• Power equipment	• See manufacturers instructions for the use of hand and portable power tools.	

BALLARD SHOP SAP

APPENDIX C

DOCUMENT COMMENTS AND RESPONSES

**REMEDIAL INVESTIGATION / FEASIBILITY STUDY
WORK PLAN FOR P4's BALLARD, HENRY AND ENOCH
VALLEY MINES**

**COMPILED A/T COMMENTS AND P4's RESPONSES
ON THE BALLARD SHOP INVESTIGATION SAP**

MAY 2011

Prepared by:
MWH AMERICAS, INC.

Prepared for:
P4 PRODUCTION, LLC



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
IDAHO OPERATIONS OFFICE
1435 N. Orchard St.
Boise, Idaho 83706

March 15, 2011

Barry Koch
Special Projects Lead – Mining
Monsanto Company
P.O. Box 816
Soda Springs, Idaho 83276

Re: Comments on *Ballard Mine Shop Investigation Sampling and Analysis Plan Revision 0 Draft*, prepared for P4 Production by MWH, February 2011.

Dear Mr. Koch,

The Agencies and Tribes (A/T) have reviewed the above referenced deliverable submitted by P4. This work product was developed pursuant to the 2009 RI/FS Settlement Agreement. Our comments are enclosed.

We will be available to discuss this matter, either by conference call, or as necessary during subject-specific meetings. I can be reached at 208-378-5763 or electronically at tomten.dave@epa.gov.

Sincerely,

//s//

Dave Tomten
Remedial Project Manager

Enclosure

cc: Cary Faulk, MWH (electronic version only)
Vance Drain, MWH (electronic version only)
Mike Rowe, IDEQ
Mary Kaufman, FS
Jim Alexander, USDA
Forest Service - Enoch Valley Site Record
Jeff Cundick, BLM
Sandi Fisher, US FWS

Kelly Wright, Shoshone Bannock Tribes
Susan Hanson (for the tribes)
Colleen O'Hara, BLM (electronic version only)
Eldine Stevens, BIA (electronic version only)
Tim Mosko, CH2MHill (electronic version only)
Sherri Clark, FS (electronic version only)
Charles Allbritton, EPA Records Center (electronic version only)

Mine Shop Investigation Sampling and Analysis Plan Revision 0 Draft
 March 15, 2011

General Comments, SAP

It is not clear whether the subject document was intended as a stand-alone SAP or an addendum to the parent QAPP and QAPP addendum. (It appears intended as a stand-alone document, but with several references to the parent QAPP). As such, it was not always clear which of the 24 elements (identified in the EPA G-5 guidance) are new, and which refer back to the parent documents. To address this concern, to avoid problems with version control, and to ensure that all QAPP elements are covered, the Ballard Shop QAPP should be reorganized to follow the structure recommended in the G-5 guidance. For each element, there should be either a clear reference to a section of the parent QAPP/QAPP Addendum, or presentation of new information. This approach should minimize any confusion or ambiguity regarding completeness and project direction. Furthermore, this approach should be followed for other upcoming SAPs (e.g., P4's radiological SAP). A recent example where the A/T has approved a QAPP that is in general conformance with the element headings in the G-5 guidance is the *Supplemental Mine Waste Rock Dump and Facility Soil and Vegetation Characterization SAP*, dated August 2009.

Based on the SAP, human health and possibly livestock appear to be on the only potential receptors at risk for the shop area. The SAP should specify why ecological receptors are not evaluated.

Specific Comments, SAP

Section 1.1, page 1-3, 1st complete paragraph. The editorial content in this paragraph is not appropriate to the SAP. Regardless of the perceived likelihood that any residual contamination will result in further remedial action or that potential contamination is minor compared to the Mine as a whole, the fact remains that potential organic contamination at the Ballard Shop is a data gap that needs evaluation to complete the RI/FS. A more appropriate place for this type of opinion/editorial is in RTCs to agency's comments. The editorial content in the following paragraph should be deleted or revised.

“However, because any hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining, if any contaminants of potential concern (COPCs) could be found at all. In addition, the risk posed by this area, even if hydrocarbons, solvents, or PCBs were detected, likely is relatively minor when compared to the possible risks associated with the overall Ballard Site. However, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area.”

Section 2.2.1. Pages 2.and 3, last complete paragraph. The description for the transformers does not note if they still contain PCB oil or have been replaced or upgraded

with non-PCB oil. The text should summarize their PCB history, if that information is available.

Section 2.2.5, Page 2-8, 1st complete paragraph. The SAP appears to say that the second soil sample for laboratory analysis will be collected from the portion of the split spoon sample with the highest field reading. The document should describe how the soil core will be screened. For example, will it be screened using headspace measurements or some other means of PID/FID measurement? The presence of soil staining and odors should also be included as field parameters that may be used to determine where soil samples are collected for laboratory analysis.

Section 2.2.5, Page 2-8, 2nd complete paragraph. The SAP text infers that soils between 10 feet and groundwater will not be logged or sampled. However, Section 3.2.2, page 3-6 of the FSP states that at SB-3 “. . .soils will be sampled continuously from ground surface to groundwater. . .” The two documents should be reconciled in this regard. Furthermore, the SAP and FSP should specify that soil sampling will continue at depth if field observations indicate contamination extends beyond 10 feet. For example, if the third sample from the 9-10 feet bgs interval exhibits significant evidence of downward migration of contaminants (either by PID/FID measurements, odors, or staining), then deeper soil samples should be logged and collected for analysis of organic COPCs. This should continue down to groundwater, as warranted by field screening, at a minimum interval of 5-feet.

Table 2-1, Step 1, 2nd paragraph, last sentence. The referenced sentence states:

“In order to provide chemical evidence that a possible release has either not impacted the environment or is not currently present at levels of concern at the Ballard Mine Shop, groundwater and soil samples from around the Ballard Mine Shop are proposed to fill this data gap based on the lack of specific data.”

To paraphrase, this sentence appears to say that the purpose of this study is to confirm the foregone conclusion that there is not a significant release at the Ballard Shop. Whereas, the purpose is to characterize existing levels of potential organic contamination in soil and groundwater and determine if they are above levels of concern, as has been stated in Step 2. The sentence should be deleted from Step 1 because it is not a statement the problem.

Table 2-1, Step 3. The following items should be added to the list of information sources for Step 3.

- 1991 UST closure investigation reports, documentation, and associated agency correspondence
- Soil and groundwater sample data to define the nature and extent and magnitude of potential releases.

Table 2-1, Step 4. The vertical boundary for soil should be the depth to groundwater to address potential releases that may extend down to groundwater, assuming DNAPL is not indicated.

Table 2-1, Step 5. As stated, the analytical approach is somewhat ambiguous and may be too open for interpretation of results. Conversely, this is essentially a screening effort, thus P4 is not presently being asked to collect sufficient sample numbers for statistical comparisons (e.g., 95% UCLs) for decision making. To better represent and qualify the analytical approach, the text in Step 5 should be revised as follows:

- The first sentence of each study question should be revised to replace the phrase “. . . above a level of concern. . .” with “. . .above risk-based screening benchmarks. . .” to make the statement consistent with the input identified in Step 3.
- The second sentence of each study question should be revised to replace the phrase “. . . data do not show impacts above a level of concern. . .” with “. . . data do not show significant impacts above risk-based screening benchmarks. . .”

Table 2-1, Step 7, 2nd sentence. Replace the word “suggests” with the word “shows” or provide another appropriate descriptive revision of the sentence. The A/T recognizes the subjectivity involved in potential decisions regarding the need for further evaluation of the sampling design, however, the word “suggests” is too vague for basing decisions.

Figure 2-2. Add an estimated groundwater flow direction arrow to the figure.

Editorial Comments, SAP

Be consistent in the use of groundwater or ground water. It appears that groundwater predominates.

Section 1.0, page 1-1, paragraph 1, line 4. Change *preformed* to “performed.”

Section 2.2.1, page 2-3, paragraph 1, line 6. Change *it* to “its.”

Section 2.2.5, page 2-9, paragraph 4 (last), line 6. Delete *of*.

Table 2-1, Step 7, line 2. Insert “are” between *operations* and *presented*.

General Comments, Appendix A

When reorganizing the QAPP consistent with the G-5 guidance, place the performance criteria (e.g., performance criteria tables and the detection limit comparison tables) such that they follow the DQOs, per the G-5 guidance.

For analytes where project criteria are lower than lab detection limits, a discussion on how these results will be used in the final evaluation should be added to the SAP.

The level of effort and information provided for the organic specs should be the same as for other constituents addressed in the QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. If P4 wishes further clarification on this issue, the A/T believes that this would be best handled in a conference call that includes A/T and P4 data quality expert staff. Furthermore, at P4's discretion, these specs may be submitted as an addendum to the QAPP/QAPP Addendum rather than just for the Ballard Shop project so they can be used for other future work, as needed. If P4 wishes to amend the QAPP/QAPP Addendum with these specs or any other information, the exact means and process to do so should first be agreed to by the A/T in order to ensure appropriate version control.

Specific Comments, Appendix A

Section 2.0, page 2-1, 1st paragraph. The same comment regarding editorial comment as made above for Section 1.1, page 1-3, 1st complete paragraph of the SAP applies here in the FSP, as well.

Section 3.1.1., page 3-2, 1st full paragraph. For PCBs, sample depths are not specified. The text should specify sample depths and describe contingency sampling at depth if field data indicate that a release extends significantly beyond the first few feet of surface soils.

Section 3.2.2., page 3-5, 2nd full paragraph. Similar to a previous comment on field screening soil samples, the text should describe how the split spoon samples will be screened, e.g., using headspace or some other means.

Section 3.2.2, page 3-7. The temporary well installation description indicates that wells will not be developed. Explain the rationale for not developing the wells or provide a description of the development methods.

Sections 4.3 and 4.4, tables 4.8, 4.9, 4.10. The analytical and data validation specs introduced here are not consistent with the specs for other constituents in the parent QAPP and QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. As noted above under general comments, if new method specific specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

Section 4.5 and Appendix B. The validation level descriptions and the proposed level of effort are not consistent with the specs and level of effort agreed to in the parent addendum. Consistent with the previous validation efforts since the development of the QAPP Addendum, all data need to be 'validated' and flagged by reviewing all QC summary data for all QC parameters (to include initial calibration, continuing calibration,

tuning, internal standards, interference checks, serial dilutions, etc.). Additionally 10% of the data needs to be reviewed for raw data. EPA data validation functional guidance should be used for reviewing and flagging the data. As noted above under general comments, if new method specific data validation specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

Editorial Comments, Appendix A

Section 3.1.2, page 3-3, paragraph 1, line 3. Delete *of*.

Section 3.2.2, page 3-6, paragraph 4 (last), line 3. Delete *than*.

Section 3.2.2, page 3-7, paragraph 3, line 1. Delete *of*.

Section 3.2.3, page 3-8, paragraph 4, line 2. Delete *in*.

Section 3.2.5, page 3-10, paragraph 2, line 2. Delete first *type of*.

A/T's March 15, 2011 Comments on P4 Production, LLC *Ballard Mine Shop Investigation Sampling and Analysis Plan - Revision 0 Draft* (dated February 24, 2011) and P4's Responses

General Comments (GC) on Ballard Mine Shop SAP

GC #1 - It is not clear whether the subject document was intended as a stand-alone SAP or an addendum to the parent QAPP and QAPP addendum. (It appears intended as a stand-alone document, but with several references to the parent QAPP). As such, it was not always clear which of the 24 elements (identified in the EPA G-5 guidance) are new, and which refer back to the parent documents. To address this concern, to avoid problems with version control, and to ensure that all QAPP elements are covered, the Ballard Shop QAPP should be reorganized to follow the structure recommended in the G-5 guidance. For each element, there should be either a clear reference to a section of the parent QAPP/QAPP Addendum, or presentation of new information. This approach should minimize any confusion or ambiguity regarding completeness and project direction. Furthermore, this approach should be followed for other upcoming SAPs (e.g., P4's radiological SAP). A recent example where the A/T has approved a QAPP that is in general conformance with the element headings in the G-5 guidance is the *Supplemental Mine Waste Rock Dump and Facility Soil and Vegetation Characterization* SAP, dated August 2009.

P4 Response: *Appendix A of the SAP (the FSP/QAPP) has been revised to address the concerns identified by the A/T, and should be considered a stand-alone FSP/QAPP. The following five sections have been added: Section 3.3 (Training Requirements), Section 3.4 (Documentation and Records Requirements), Section 4.6 (Data Management), Section 4.7 (Assessment and Response Actions), and Section 4.8 (Reports to Management). Table RTC-1 attached to this response to comments is provided as a cross reference to ensure that all 24 QAPP elements have been addressed. The Waste Rock Dump SAP will be used as the template for future P4 SAPs.*

GC #2 - Based on the SAP, human health and possibly livestock appear to be on the only potential receptors at risk for the shop area. The SAP should specify why ecological receptors are not evaluated.

P4 Response: *The Ballard Shop area is largely industrial and devoid of any forage for wildlife and of any flowing or standing water that would be attractive to ecological receptors or would provide habitat. The Conceptual Model text in Section 2.2.2 has been modified to express this.*

Specific Comments (SC) on the Ballard Mine Shop SAP

SC#1 - Section 1.1, page 1-3, 1st complete paragraph. The editorial content in this paragraph is not appropriate to the SAP. Regardless of the perceived likelihood that any residual contamination will result in further remedial action or that potential contamination is minor compared to the Mine as a whole, the fact remains that potential organic contamination at the Ballard Shop is a data gap that needs evaluation to complete the RI/FS. A more appropriate place for this type of opinion/editorial is in RTCs to agency's comments. The editorial content in the following paragraph should be deleted or revised.

“However, because any hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining, if any contaminants of potential concern (COPCs) could be found at all. In addition, the risk posed by this area, even if hydrocarbons, solvents, or PCBs were detected, likely is relatively minor when compared to the possible risks associated with the overall Ballard Site. However, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area.”

P4 Response: *This paragraph where that text was presented was revised as follows:*

“During the active operation period, there may have been incidental spills/ leaks of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining.”

SC#2 - Section 2.2.1. Pages 2.and 3, last complete paragraph. The description for the transformers does not note if they still contain PCB oil or have been replaced or upgraded with non-PCB oil. The text should summarize their PCB history, if that information is available.

P4 Response: *There has been some limited sampling of the large transformers on the pad in front of the mine shop (i.e., to the west). As of 1995, the PCB levels were very low to not detectable in the transformer oil. However, there is no information on the three elevated transformers located on the south side of the shop. The soil sampling that is proposed should tell us if oils containing PCBs ever leaked from these transformers in either of these locations. This information has been added to Section 2.2.1.*

SC#3 - Section 2.2.5, Page 2-8, 1st complete paragraph. The SAP appears to say that the second soil sample for laboratory analysis will be collected from the portion of the split spoon sample with the highest field reading. The document should describe how the soil core will be screened. For example, will it be screened using headspace measurements or some other means of PID/FID measurement? The presence of soil staining and odors should also be included as field parameters that may be used to determine where soil samples are collected for laboratory analysis.

P4 Response: *Upon retrieval from the borehole, the split spoon sampler will be laid on the vise and opened by the driller or geologist. The sample then will be cut open using a stainless steel knife to log the soil core and to collect PID/FID measurements by drawing the air in immediately above the sample face. If there is significant contamination, the PID will indicate it by elevated readings. The geologist will select the interval exhibiting the highest PID readings along the soil core for sample collection. However, if there are no significant readings, as you suggest, they will rely on staining or smell to define the appropriate sample interval. Should there be no PID readings, staining, or smell then the interval nearest the bottom of that sample will be used for our selected soil sample. Text has been added to Section 2.2.5 and in Section 3.2.2. of the FSP to further clarify this approach.*

SC#4 - Section 2.2.5, Page 2-8, 2nd complete paragraph. The SAP text infers that soils between 10 feet and groundwater will not be logged or sampled. However, Section 3.2.2, page 3-6 of the FSP states that at SB-3 “. . .soils will be sampled continuously from ground surface to groundwater. . .” The two documents should be reconciled in this regard. Furthermore, the SAP and FSP should specify that soil sampling will continue at depth if field observations indicate contamination extends beyond 10 feet. For example, if the third sample from the 9-10 feet bgs interval exhibits significant evidence of downward migration of contaminants (either by PID/FID measurements, odors, or staining), then deeper soil samples should be logged and collected for analysis of organic COPCs. This should continue down to groundwater, as warranted by field screening, at a minimum interval of 5-feet.

P4 Response: *If contamination is detected in the 9-10 foot sample, then as suggested P4 will continue to collect 2-foot long split spoon soil samples at 5 foot interval for logging and field screening purposes using PID readings, odor and staining, as suggested. However, only 1 additional soil sample will be collected to define the lower limits (extent) of contamination and that sample will be collected from the first 5 foot interval that has no PID readings, or staining or odor. Text has been added to Section 2.2.5 and in Section 3.2.2. of the FSP to further clarify this approach.*

SC#5 - Table 2-1, Step 1, 2nd paragraph, last sentence. The referenced sentence states:

“In order to provide chemical evidence that a possible release has either not impacted the environment or is not currently present at levels of concern at the Ballard Mine Shop, groundwater and soil samples from around the Ballard Mine Shop are proposed to fill this data gap based on the lack of specific data.”

To paraphrase, this sentence appears to say that the purpose of this study is to confirm the foregone conclusion that there is not a significant release at the Ballard Shop. Whereas, the purpose is to characterize existing levels of potential organic contamination in soil and groundwater and determine if they are above levels of concern, as has been stated in Step 2. The sentence should be deleted from Step 1 because it is not a statement the problem.

P4 Response: *Agreed. P4 has removed that statement because the goals of the study are defined in Step 2, as you suggest.*

SC#6 - Table 2-1, Step 3. The following items should be added to the list of information sources for Step 3.

- 1991 UST closure investigation reports, documentation, and associated agency correspondence
- Soil and groundwater sample data to define the nature and extent and magnitude of potential releases.

P4 Response: *This information has been added to Table 2-1, Step 3, as requested.*

SC#7 - Table 2-1, Step 4. The vertical boundary for soil should be the depth to groundwater to address potential releases that may extend down to groundwater, assuming DNAPL is not indicated.

P4 Response: *Please refer to our response to SC#4 above. The total planned depth of soil investigation is 10 feet below ground surface based on the needs of the risk assessment, however additional soil samples will be collected if the sample interval at 10 feet indicates contamination. This caveat has been added to Step 4.*

SC#8 - Table 2-1, Step 5. As stated, the analytical approach is somewhat ambiguous and may be too open for interpretation of results. Conversely, this is essentially a screening effort, thus P4 is not presently being asked to collect sufficient sample numbers for statistical comparisons (e.g., 95%UCLs) for decision making. To better represent and qualify the analytical approach, the text in Step 5 should be revised as follows:

- The first sentence of each study question should be revised to replace the phrase "... above a level of concern. . ." with "...above risk-based screening benchmarks. . ." to make the statement consistent with the input identified in Step 3.
- The second sentence of each study question should be revised to replace the phrase "... data do not show impacts above a level of concern. . ." with "... data do not show significant impacts above risk-based screening benchmarks. . ."

P4 Response: *These principal study questions under the analytical approach in Step 5 have been revised as suggested.*

SC#9- Table 2-1, Step 7, 2nd sentence. Replace the word "suggests" with the word "shows" or provide another appropriate descriptive revision of the sentence. The A/T recognizes the subjectivity involved in potential decisions regarding the need for further evaluation of the sampling design, however, the word "suggests" is too vague for basing decisions.

P4 Response: *The word "suggests" has been replaced by "indicates".*

SC#10 - Figure 2-2. Add an estimated groundwater flow direction arrow to the figure.

P4 Response: *A probable range of groundwater flow direction was added to Figure 2-2.*

Editorial Comments on the Ballard Mine SAP

EC #1 - Be consistent in the use of groundwater or ground water. It appears that groundwater predominates.

P4 Response: *Groundwater will be one word throughout the document. During the search of the text, only one incidence was found where it was in the two word form.*

EC #2 - Section 1.0, page 1-1, paragraph 1, line 4. Change *preformed* to "performed."

P4 Response: *Agreed*

EC #3 - Section 2.2.1, page 2-3, paragraph 1, line 6. Change it to "its."

P4 Response: *Agreed*

EC #4 - Section 2.2.5, page 2-9, paragraph 4 (last), line 6. Delete of.

P4 Response: *Agreed*

EC #5 - Table 2-1, Step 7, line 2. Insert "are" between operations and presented.

P4 Response: *Agreed.*

General Comments on Appendix A – FSP/QAPP

A#1 - When reorganizing the QAPP consistent with the G-5 guidance, place the performance criteria (e.g., performance criteria tables and the detection limit comparison tables) such that they follow the DQOs, per the G-5 guidance.

P4 Response: *The G-5 guidance specifies inclusion of the measurement performance criteria in Step 7 of the DQO process. Therefore, the last paragraph of Section 2.1 and Step 7 in Table 2-1 (DQOs) have been revised to reference FSP/QAPP Table 4-5, the project performance measurement criteria, and FSP/QAPP Tables 4-6 and 4-7, the table presenting comparisons of laboratory detection limits to the human health screening levels for soil and groundwater, respectively.*

A#2 - For analytes where project criteria are lower than lab detection limits, a discussion on how these results will be used in the final evaluation should be added to the SAP.

P4 Response: *The following paragraph has been added to the end of the text in Section 5.3 of the FSP/QAPP, "Table 4-5 notes that there are the method detection limits for several target compounds are greater than the human health screening levels for soil and groundwater. The specific compounds are identified with footnote "c" on Table 4-6 for soil (four SVOCs) and on Table 4-7 for groundwater (seven VOCs and 11 SVOCs). The uncertainty related to this will be addressed as part of the human health risk assessment."*

A#3 - The level of effort and information provided for the organic specs should be the same as for other constituents addressed in the QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. If P4 wishes further clarification on this issue, the A/T believes that this would be best handled in a conference call that includes A/T and P4 data quality expert staff. Furthermore, at P4's discretion, these specs may be submitted as an addendum to the QAPP/QAPP Addendum rather than just for the Ballard Shop project so they can be used for other future work, as needed. If P4 wishes to amend the QAPP/QAPP Addendum with

these specs or any other information, the exact means and process to do so should first be agreed to by the A/T in order to ensure appropriate version control.

P4 Response: *Section 3.4.4 has been added to provide specific detail for the laboratory hard-copy data deliverables for VOCs, SVOCs, and PCBs. The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the organic methods as has been provided in the QAPP Addendum for inorganic constituents. The data validation report templates (now provided in Appendix C of the FSP/QAPP) have been revised to reference the FSP/QAPP tables. Please note that the target compounds list is too extensive to concisely summarize the control limits in the text of the templates. P4 does not anticipate future investigations for organic constituents and prefers to provide the sampling and analytical criteria for these organic methods in this stand-alone SAP.*

Specific Comments, Appendix A

A#4 - Section 2.0, page 2-1, 1st paragraph. The same comment regarding editorial comment as made above for Section 1.1, page 1-3, 1st complete paragraph of the SAP applies here in the FSP, as well.

P4 Response: *The first paragraph in section 2 of the FSP now reads:*

“This section provides brief background information related to the Ballard Mine Shop investigation. Additional program background and objective details are provided in Section 1.1 of the SAP and the RI/FS Work Plan. Because this facility was operated as a maintenance shop for heavy trucks and mining equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines there may have been incidental spills of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining.”

A#5 - Section 3.1.1., page 3-2, 1st full paragraph. For PCBs, sample depths are not specified. The text should specify sample depths and describe contingency sampling at depth if field data indicate that a release extends significantly beyond the first few feet of surface soils.

P4 Response: *Section 3.2 “Sample Collection Procedure” discusses where and how the soil samples will be collected during the Ballard Mine Shop investigation. The final paragraph in Section 3.2.2 indicates that two soil samples will be collected at each PCBs boring location; the first below the slag/native soil interface and the second at 4-5 feet below the existing ground surface. Additional information has been added to Section 3.2.2 (Soil Investigation (PCBs)) to indicate the boreholes will be extended if visual contamination or odors occur at the second sample depth. This section now reads:*

“Soil Investigation (PCBs). *Shallow soil samples also will be collected in the two identified transformer locations and analyzed for PCBs. Soil borings SB-5 and SB-6 will be located next to the identified transformer areas to the west and south of the shop building as depicted on Figure 3-1. The HSA drill rig will be utilized to advance these two soil borings within the alluvial material to the required depth (refer to SOP-1). Soil samples will be collected with a CME (or similar) split barrel sampling system or split-spoon samplers. Samples will be collected at the native soil interface, which is assumed to be*

approximately six to 12 inches bgs. A second sample interval will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil).

Should visual contamination or odors be detected in the second sample interval, then the boreholes will be continuously cored until no contamination indicators are observed or groundwater is reached. A third and final soil sample then will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.

The soil samples will be collected with a clean stainless steel spoon or scoop and placed in an appropriately-sized container as provided by the laboratory. Sampled soil intervals will be logged in general accordance with USCS protocol. The soil samples will be analyzed for PCBs according to the methods described on Table 3-2 and in Section 4.3."

A#6 - Section 3.2.2., page 3-5, 2nd full paragraph. Similar to a previous comment on field screening soil samples, the text should describe how the split spoon samples will be screened, e.g., using headspace or some other means.

P4 Response: *Section 3.2 has been revised to indicate how each soil core interval from the split spoon sampler will be screened in the field. Please refer to the revisions in Section 3.2.2.*

A#7 - Section 3.2.2, page 3-7. The temporary well installation description indicates that wells will not be developed. Explain the rationale for not developing the wells or provide a description of the development methods.

P4 Response: *The temporary wells will not be developed because we are planning to abandon them once the groundwater sampling and water level collection activities are completed. As described, we are planning to collect grab groundwater samples from each of these boreholes that may be turbid. As a result, these samples would indicate "worse case" contaminant conditions if contaminants are present because the sediments in the groundwater, in addition to the groundwater, could contain the constituents of potential concern. However, these temporary wells will be developed if contamination is detected above relevant benchmarks and they are need for further monitoring at the site. Well development procedures will be described in a subsequent addendum to this SAP if it is necessary.*

A#8 - Sections 4.3 and 4.4, tables 4.8, 4.9, 4.10. The analytical and data validation specs introduced here are not consistent with the specs for other constituents in the parent QAPP and QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. As noted above under general comments, if new method specific specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: *Please refer to the response to Comment A#3 above regarding the addition of Section 3.4.4 to address specific requirement for hard-copy laboratory deliverables for VOCs, SVOCs, and PCBs and existing analytical specificity provided in Tables 4-8 through 4-13. Section 4.5 has been revised to provide additional "Reason Codes" for organic methods (and these reason codes have been added to the data validation report templates). The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the validation of organic data as has been provided in the QAPP*

Addendum for inorganic data. The data validation templates have been revised to include additional level of detail.

A#9 - Section 4.5 and Appendix B. The validation level descriptions and the proposed level of effort are not consistent with the specs and level of effort agreed to in the parent addendum. Consistent with the previous validation efforts since the development of the QAPP Addendum, all data need to be 'validated' and flagged by reviewing all QC summary data for all QC parameters (to include initial calibration, continuing calibration, tuning, internal standards, interference checks, serial dilutions, etc.). Additionally 10% of the data needs to be reviewed for raw data. EPA data validation functional guidance should be used for reviewing and flagging the data. As noted above under general comments, if new method specific data validation specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: *With revisions to the Reason Codes (see response to Comment A#8), revised Section 4.5 provides the equivalent level of detail for the validation of organic data as has been provided in the QAPP Addendum for inorganic data. The data validation templates (now provided in Appendix C) have been revised to provide additional detail.*

Editorial Comments on Appendix A

A#10 - Section 3.1.2, page 3-3, paragraph 1, line 3. Delete *of*.

P4 Response: *Agreed.*

A#11 - Section 3.2.2, page 3-6, paragraph 4 (last), line 3. Delete *than*.

P4 Response: *Agreed.*

A#12 - Section 3.2.2, page 3-7, paragraph 3, line 1. Delete *of*.

P4 Response: *Agreed.*

A#13 - Section 3.2.3, page 3-8, paragraph 4, line 2. Delete *in*.

P4 Response: *Agreed.*

A#14 - Section 3.2.5, page 3-10, paragraph 2, line 2. Delete first *type of*.

P4 Response: *Agreed.*

**Table RTC-1
QAPP Elements**

Item No.	QAPP Element	Document Location or Addressed Herein
Group A	<i>Project Management</i>	
A.1	Title and Approval Sheet	FSP/QAPP Approval Page
A.2	Table of Contents	SAP and FSP/QAPP TOCs
A.3	Distribution List	This SAP is an appendix the RI/FS Work Plan; the distribution list is presented in Section 1.3.3 of the Work Plan.
A.4	Project/Task Organization	SAP Section 1.0 and FSP/QAPP Section 5.1
A.5	Problem Definition/Background	SAP Section 1.1
A.6	Project/Task Description	SAP Section 2.2.2
A.7	Quality Objectives and Criteria for Measurement Data	SAP Section 2.1 and Table 2-1; FSP/QAPP Table 4-5
A.8	Special Training Needs/Certifications	FSP/QAPP Section 3.3
A.9	Documents and Records	FSP/QAPP Section 3.4
Group B	<i>Data Generation and Acquisition</i>	
B.1	Sampling Process Design	SAP Section 2.2.4
B.2	Sampling Methods	FSP/QAPP Section 3.2.2
B.3	Sample Handling and Custody	FSP/QAPP Sections 4.1 and 3.4.3
B.4	Analytical Methods	FSP/QAPP Section 4.3
B.5	Quality Control	FSP/QAPP Section 4.4
B.6	Instrument/Equipment Testing, Inspection, and Maintenance	FSP/QAPP Table 4-14
B.7	Instrument/Equipment Calibration and Frequency	FSP/QAPP Tables 4-8, 4-9, and 4-10
B.8	Inspection/Acceptance of Supplies and Consumables	FSP/QAPP Section 4.2
B.9	Non-direct Measurements	Screening values listed on FSP/QAPP Tables 4-6 and 4-7
B.10	Data Management	FSP/QAPP Section 4.6
Group C	<i>Assessment and Oversight</i>	
C.1	Assessment and Response Actions	FSP/QAPP Section 4.7
C.2	Reports to Management	FSP/QAPP Section 4.8
Group D	<i>Data Validation and Usability</i>	
D.1	Data Review, Verification, and Validation	FSP/QAPP Section 4.5
D.2	Verification and Validation Methods	FSP/QAPP Section 4.5
D.3	Reconciliation and User Requirements	FSP/QAPP Section 4.5 and 5.3



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
IDAHO OPERATIONS OFFICE
1435 N. Orchard St.
Boise, Idaho 83706

March 29, 2011

Barry Koch
Special Projects Lead – Mining
Monsanto Company
P.O. Box 816
Soda Springs, Idaho 83276

Re: Comments on P4's Response to Comment Document on Ballard Mine Shop Investigation SAP, prepared for P4 Production by MWH, March 22, 2011.

Dear Mr. Koch,

The Agencies and Tribes (A/T) have reviewed the above referenced deliverable submitted by P4. This work product was developed pursuant to the 2009 RI/FS Settlement Agreement. Our comments on the response to comment document are enclosed.

We will be available to discuss this matter, either by conference call, or as necessary during subject-specific meetings. If no further discussion is needed, it's our understanding that you will submit an electronic version of the revised deliverable for our final review. I can be reached at 208-378-5763 or electronically at tomten.dave@epa.gov.

Sincerely,

//s//

Dave Tomten
Remedial Project Manager

Enclosure

cc: Cary Faulk, MWH (electronic version only)
Vance Drain, MWH (electronic version only)
Mike Rowe, IDEQ
Mary Kaufman, FS



Jim Alexander, USDA
Forest Service - Enoch Valley Site Record
Jeff Cundick, BLM
Sandi Fisher, US FWS
Kelly Wright, Shoshone Bannock Tribes
Susan Hanson (for the tribes)
Colleen O'Hara, BLM (electronic version only)
Eldine Stevens, BIA (electronic version only)
Tim Mosko, CH2MHill (electronic version only)
Sherri Clark, FS (electronic version only)
Charles Allbritton, EPA Records Center (electronic version only)

Comments on P4's Response to Comment Document on Ballard Mine Shop Investigation SAP, prepared for P4 Production by MWH, March 22, 2011.
March 29, 2011

A/T Comment GC #1: It is not clear whether the subject document was intended as a stand-alone SAP or an addendum to the parent QAPP and QAPP addendum. (It appears intended as a stand-alone document, but with several references to the parent QAPP). As such, it was not always clear which of the 24 elements (identified in the EPA G-5 guidance) are new, and which refer back to the parent documents. To address this concern, to avoid problems with version control, and to ensure that all QAPP elements are covered, the Ballard Shop QAPP should be reorganized to follow the structure recommended in the G-5 guidance. For each element, there should be either a clear reference to a section of the parent QAPP/QAPP Addendum, or presentation of new information. This approach should minimize any confusion or ambiguity regarding completeness and project direction. Furthermore, this approach should be followed for other upcoming SAPs (e.g., P4's radiological SAP). A recent example where the A/T has approved a QAPP that is in general conformance with the element headings in the G-5 guidance is the *Supplemental Mine Waste Rock Dump and Facility Soil and Vegetation Characterization SAP*, dated August 2009.

P4 Response: Appendix A of the SAP (the FSP/QAPP) has been revised to address the concerns identified by the A/T, and should be considered a stand-alone FSP/QAPP. The following five sections have been added: Section 3.3 (Training Requirements), Section 3.4 (Documentation and Records Requirements), Section 4.6 (Data Management), Section 4.7 (Assessment and Response Actions), and Section 4.8 (Reports to Management). Table RTC-1 attached to this response to comments is provided as a cross reference to ensure that all 24 QAPP elements have been addressed. The Waste Rock Dump SAP will be used as the template for future P4 SAPs.

***A/T Response.** Please include the cross reference table that you have developed in the document. In addition, we agree that the Waste Rock Dump SAP is a good template for future SAPs as it closely follows the organization recommended in the G-5 guidance. Following the standard template promotes technical consistency, and is the most economical approach to meeting the G-5 requirements.*

A/T Comment SC#4 - Section 2.2.5, Page 2-8, 2nd complete paragraph. The SAP text infers that soils between 10 feet and groundwater will not be logged or sampled. However, Section 3.2.2, page 3-6 of the FSP states that at SB-3 “. . .soils will be sampled continuously from ground surface to groundwater. . .” The two documents should be reconciled in this regard. Furthermore, the SAP and FSP should specify that soil sampling will continue at depth if field observations indicate contamination extends beyond 10 feet. For example, if the third sample from the 9-10 feet bgs interval exhibits significant evidence of downward migration of contaminants (either by PID/FID measurements, odors, or staining), then deeper soil samples should be logged and collected for analysis of organic COPCs. This should continue down to groundwater, as warranted by field screening, at a minimum interval of 5-feet.

P4 Response: If contamination is detected in the 9-10 foot sample, then as suggested P4 will continue to collect 2-foot long split spoon soil samples at 5 foot interval for logging and field screening purposes using PID readings, odor and staining, as suggested. However, only 1 additional soil sample will be collected to define the lower limits (extent) of contamination and that sample will be collected from the first 5 foot interval that has no PID readings, or staining or odor. Text has been added to Section 2.2.5 and in Section 3.2.2. of the FSP to further clarify this approach.

***A/T Response.** On March 28, 2011, P4 provided the following clarification, which is acceptable to the A/T.*

“Based on a significant hit, as recorded above background by the field instrumentation (i.e., PID /FID) at the 5 foot bgs interval, a third soil sample would be collected at a depth of nine to 10 feet bgs and screened as described above before soil sample collection. Should significant contamination be detected in the 10 foot interval, then the borehole will be continuously cored until no PID readings, visual staining, or odors are observed or groundwater is encountered. A fourth and final soil sample will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.”

Furthermore, as noted by P4 in our March 28, 2011 biweekly project conference call, this SAP is screen for potential historic releases at the Ballard Shop. If results suggest significant release of contaminants, additional characterization may be necessary.

A/T Comment A#3: The level of effort and information provided for the organic specs should be the same as for other constituents addressed in the QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. If P4 wishes further clarification on this issue, the A/T believes that this would be best handled in a conference call that includes A/T and P4 data quality expert staff. Furthermore, at P4’s discretion, these specs may be submitted as an addendum to the QAPP/QAPP Addendum rather than just for the Ballard Shop project so they can be used for other future work, as needed. If P4 wishes to amend the QAPP/QAPP Addendum with these specs or any other information, the exact means and process to do so should first be agreed to by the A/T in order to ensure appropriate version control.

P4 Response: Section 3.4.4 has been added to provide specific detail for the laboratory hard-copy data deliverables for VOCs, SVOCs, and PCBs. The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the organic methods as has been provided in the QAPP Addendum for inorganic constituents. The data validation report templates (now provided in Appendix C of the FSP/QAPP) have been revised to reference the FSP/QAPP tables. Please note that the target compounds list is too extensive to concisely summarize the control limits in the text of the templates. P4 does not anticipate future investigations for organic constituents and prefers to provide the sampling and analytical criteria for these organic methods in this stand-alone SAP.

***A/T Response:** The validation templates in the Addendum contained the final report to be used by LDC (or any other validator). Please confirm that report is being provided for the Ballard Shop study.*

A/T Comment A#8 - Sections 4.3 and 4.4, tables 4.8, 4.9, 4.10. The analytical and data validation specs introduced here are not consistent with the specs for other constituents in the parent QAPP and QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. As noted above under general comments, if new method specific specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: Please refer to the response to Comment A#3 above regarding the addition of Section 3.4.4 to address specific requirement for hard-copy laboratory deliverables for VOCs, SVOCs, and PCBs and existing analytical specificity provided in Tables 4-8 through 4-13. Section 4.5 has been revised to provide additional “Reason Codes” for organic methods (and these reason codes have been added to the data validation report templates). The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the validation of organic data as has been provided in the QAPP Addendum for inorganic data. The data validation templates have been revised to include additional level of detail.

***A/T Response:** To clarify, the data validation requirements/specs and data validation report templates need to be identical to the Addendum with method specific elements.*

A/T Comment A#9 - Section 4.5 and Appendix B. The validation level descriptions and the proposed level of effort are not consistent with the specs and level of effort agreed to in the parent addendum. Consistent with the previous validation efforts since the development of the QAPP Addendum, all data need to be ‘validated’ and flagged by reviewing all QC summary data for all QC parameters (to include initial calibration, continuing calibration, tuning, internal standards, interference checks, serial dilutions, etc.). Additionally 10% of the data needs to be reviewed for raw data. EPA data validation functional guidance should be used for reviewing and flagging the data. As noted above under general comments, if new method specific data validation specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: With revisions to the Reason Codes (see response to Comment A#8), revised Section 4.5 provides the equivalent level of detail for the validation of organic data as has been provided in the QAPP Addendum for inorganic data. The data validation templates (now provided in Appendix C) have been revised to provide additional detail.

***A/T Response:** Please see the A/T response to comment A#8, above.*

A/T's Original (March 15, 2011) and Follow-up (March 29, 2011) Comments on P4 Production, LLC Ballard Mine Shop Investigation Sampling and Analysis Plan - Revision 0 Draft (dated February 24, 2011) and P4's Responses

Deleted: March 15, 2011

General Comments (GC) on Ballard Mine Shop SAP

GC #1 - It is not clear whether the subject document was intended as a stand-alone SAP or an addendum to the parent QAPP and QAPP addendum. (It appears intended as a stand-alone document, but with several references to the parent QAPP). As such, it was not always clear which of the 24 elements (identified in the EPA G-5 guidance) are new, and which refer back to the parent documents. To address this concern, to avoid problems with version control, and to ensure that all QAPP elements are covered, the Ballard Shop QAPP should be reorganized to follow the structure recommended in the G-5 guidance. For each element, there should be either a clear reference to a section of the parent QAPP/QAPP Addendum, or presentation of new information. This approach should minimize any confusion or ambiguity regarding completeness and project direction. Furthermore, this approach should be followed for other upcoming SAPs (e.g., P4's radiological SAP). A recent example where the A/T has approved a QAPP that is in general conformance with the element headings in the G-5 guidance is the *Supplemental Mine Waste Rock Dump and Facility Soil and Vegetation Characterization SAP*, dated August 2009.

P4 Response: *Appendix A of the SAP (the FSP/QAPP) has been revised to address the concerns identified by the A/T, and should be considered a stand-alone FSP/QAPP. The following five sections have been added: Section 3.3 (Training Requirements), Section 3.4 (Documentation and Records Requirements), Section 4.6 (Data Management), Section 4.7 (Assessment and Response Actions), and Section 4.8 (Reports to Management). Table RTC-1 attached to this response to comments is provided as a cross reference to ensure that all 24 QAPP elements have been addressed. The Waste Rock Dump SAP will be used as the template for future P4 SAPs.*

A/T Follow-up Comment: Please include the cross reference table that you have developed in the document. In addition, we agree that the Waste Rock Dump SAP is a good template for future SAPs as it closely follows the organization recommended in the G-5 guidance. Following the standard template promotes technical consistency, and is the most economical approach to meeting the G-5 requirements.

P4 Follow-up Response: Agreed. The referenced table has been added as Table 1-1 of the FSP/QAPP (Appendix A of the SAP).

GC #2 - Based on the SAP, human health and possibly livestock appear to be on the only potential receptors at risk for the shop area. The SAP should specify why ecological receptors are not evaluated.

P4 Response: *The Ballard Shop area is largely industrial and devoid of any forage for wildlife and of any flowing or standing water that would be attractive to ecological receptors or would provide habitat. The Conceptual Model text in Section 2.2.2 has been modified to express this.*

Specific Comments (SC) on the Ballard Mine Shop SAP

SC#1 - Section 1.1, page 1-3, 1st complete paragraph. The editorial content in this paragraph is not appropriate to the SAP. Regardless of the perceived likelihood that any residual contamination will result in further remedial action or that potential contamination is minor compared to the Mine as a whole, the fact remains that potential organic contamination at the Ballard Shop is a data gap that needs evaluation to complete the RI/FS. A more appropriate place for this type of opinion/editorial is in RTCs to agency's comments. The editorial content in the following paragraph should be deleted or revised.

"However, because any hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining, if any contaminants of potential concern (COPCs) could be found at all. In addition, the risk posed by this area, even if hydrocarbons, solvents, or PCBs were detected, likely is relatively minor when compared to the possible risks associated with the overall Ballard Site. However, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area."

P4 Response: *This paragraph where that text was presented was revised as follows:*

"During the active operation period, there may have been incidental spills/leaks of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining."

SC#2 - Section 2.2.1. Pages 2.and 3, last complete paragraph. The description for the transformers does not note if they still contain PCB oil or have been replaced or upgraded with non-PCB oil. The text should summarize their PCB history, if that information is available.

P4 Response: *There has been some limited sampling of the large transformers on the pad in front of the mine shop (i.e., to the west). As of 1995, the PCB levels were very low to not detectable in the transformer oil. However, there is no information on the three elevated transformers located on the south side of the shop. The soil sampling that is proposed should tell us if oils containing PCBs ever leaked from these transformers in either of these locations. This information has been added to Section 2.2.1.*

SC#3 - Section 2.2.5, Page 2-8, 1st complete paragraph. The SAP appears to say that the second soil sample for laboratory analysis will be collected from the portion of the split spoon sample with the highest field reading. The document should describe how the soil core will be screened. For example, will it be screened using headspace measurements or some other means of PID/FID measurement? The presence of soil staining and odors should also be included as field parameters that may be used to determine where soil samples are collected for laboratory analysis.

P4 Response: Upon retrieval from the borehole, the split spoon sampler will be laid on the vise and opened by the driller or geologist. The sample then will be cut open using a stainless steel knife to log the soil core and to collect PID/FID measurements by drawing the air in immediately above the sample face. If there is significant contamination, the PID will indicate it by elevated readings. The geologist will select the interval exhibiting the highest PID readings along the soil core for sample collection. However, if there are no significant readings, as you suggest, they will rely on staining or smell to define the appropriate sample interval. Should there be no PID readings, staining, or smell then the interval nearest the bottom of that sample will be used for our selected soil sample. Text has been added to Section 2.2.5 and in Section 3.2.2. of the FSP to further clarify this approach.

SC#4 - Section 2.2.5, Page 2-8, 2nd complete paragraph. The SAP text infers that soils between 10 feet and groundwater will not be logged or sampled. However, Section 3.2.2, page 3-6 of the FSP states that at SB-3 "...soils will be sampled continuously from ground surface to groundwater. . ." The two documents should be reconciled in this regard. Furthermore, the SAP and FSP should specify that soil sampling will continue at depth if field observations indicate contamination extends beyond 10 feet. For example, if the third sample from the 9-10 feet bgs interval exhibits significant evidence of downward migration of contaminants (either by PID/FID measurements, odors, or staining), then deeper soil samples should be logged and collected for analysis of organic COPCs. This should continue down to groundwater, as warranted by field screening, at a minimum interval of 5-feet.

P4 Response: If contamination is detected in the 9-10 foot sample, then as suggested P4 will continue to collect 2-foot long split spoon soil samples at 5 foot interval for logging and field screening purposes using PID readings, odor and staining, as suggested. However, only 1 additional soil sample will be collected to define the lower limits (extent) of contamination and that sample will be collected from the first 5 foot interval that has no PID readings, or staining or odor. Text has been added to Section 2.2.5 and in Section 3.2.2. of the FSP to further clarify this approach.

A/T Follow-up Comment: On March 28, 2011, P4 provided the following clarification, which is acceptable to the A/T.

"Based on a significant hit, as recorded above background by the field instrumentation (i.e., PID /FID) at the 5 foot bgs interval, a third soil sample would be collected at a depth of nine to 10 feet bgs and screened as described above before soil sample collection. Should significant contamination be detected in the 10 foot interval, then the borehole will be continuously cored until no PID readings, visual staining, or odors are observed or groundwater is encountered. A fourth and final soil sample will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination."

Furthermore, as noted by P4 in our March 28, 2011 biweekly project conference call, this SAP is screen for potential historic releases at the Ballard Shop. If results suggest significant release of contaminants, additional characterization may be necessary.

P4 Follow-up Response: Agreed. Table 4-4 (Samples to be Collected) has been revised to include a potential fourth soil sample, to be collected just beneath the identified contamination or just above the water table in each boring.

SC#5 - Table 2-1, Step 1, 2nd paragraph, last sentence. The referenced sentence states:

"In order to provide chemical evidence that a possible release has either not impacted the environment or is not currently present at levels of concern at the Ballard Mine Shop, groundwater and soil samples from around the Ballard Mine Shop are proposed to fill this data gap based on the lack of specific data."

To paraphrase, this sentence appears to say that the purpose of this study is to confirm the foregone conclusion that there is not a significant release at the Ballard Shop. Whereas, the purpose is to characterize existing levels of potential organic contamination in soil and groundwater and determine if they are above levels of concern, as has been stated in Step 2. The sentence should be deleted from Step 1 because it is not a statement the problem.

P4 Response: *Agreed. P4 has removed that statement because the goals of the study are defined in Step 2, as you suggest.*

SC#6 - Table 2-1, Step 3. The following items should be added to the list of information sources for Step 3.

- 1991 UST closure investigation reports, documentation, and associated agency correspondence
- Soil and groundwater sample data to define the nature and extent and magnitude of potential releases.

P4 Response: *This information has been added to Table 2-1, Step 3, as requested.*

SC#7 - Table 2-1, Step 4. The vertical boundary for soil should be the depth to groundwater to address potential releases that may extend down to groundwater, assuming DNAPL is not indicated.

P4 Response: *Please refer to our response to SC#4 above. The total planned depth of soil investigation is 10 feet below ground surface based on the needs of the risk assessment, however additional soil samples will be collected if the sample interval at 10 feet indicates contamination. This caveat has been added to Step 4.*

SC#8 - Table 2-1, Step 5. As stated, the analytical approach is somewhat ambiguous and may be too open for interpretation of results. Conversely, this is essentially a screening effort, thus P4 is not presently being asked to collect sufficient sample numbers for statistical comparisons (e.g., 95% UCLs) for decision making. To better represent and qualify the analytical approach, the text in Step 5 should be revised as follows:

- The first sentence of each study question should be revised to replace the phrase "... above a level of concern. ." with "...above risk-based screening benchmarks. ." to make the statement consistent with the input identified in Step 3.

- The second sentence of each study question should be revised to replace the phrase "... data do not show impacts above a level of concern. ..." with "... data do not show significant impacts above risk-based screening benchmarks. ..."

P4 Response: *These principal study questions under the analytical approach in Step 5 have been revised as suggested.*

SC#9- Table 2-1, Step 7, 2nd sentence. Replace the word "suggests" with the word "shows" or provide another appropriate descriptive revision of the sentence. The A/T recognizes the subjectivity involved in potential decisions regarding the need for further evaluation of the sampling design, however, the word "suggests" is too vague for basing decisions.

P4 Response: *The word "suggests" has been replaced by "indicates".*

SC#10 - Figure 2-2. Add an estimated groundwater flow direction arrow to the figure.

P4 Response: *A probable range of groundwater flow direction was added to Figure 2-2.*

Editorial Comments on the Ballard Mine SAP

EC #1 - Be consistent in the use of groundwater or ground water. It appears that groundwater predominates.

P4 Response: *Groundwater will be one word throughout the document. During the search of the text, only one incidence was found where it was in the two word form.*

EC #2 - Section 1.0, page 1-1, paragraph 1, line 4. Change *preformed* to "performed."

P4 Response: *Agreed.*

EC #3 - Section 2.2.1, page 2-3, paragraph 1, line 6. Change it to "its."

P4 Response: *Agreed.*

EC #4 - Section 2.2.5, page 2-9, paragraph 4 (last), line 6. Delete *of*.

P4 Response: *Agreed.*

EC #5 - Table 2-1, Step 7, line 2. Insert "are" between *operations* and *presented*.

P4 Response: *Agreed.*

General Comments on Appendix A – FSP/QAPP

A#1 - When reorganizing the QAPP consistent with the G-5 guidance, place the performance criteria (e.g., performance criteria tables and the detection limit comparison tables) such that they follow the DQOs, per the G-5 guidance.

P4 Response: *The G-5 guidance specifies inclusion of the measurement performance criteria in Step 7 of the DQO process. Therefore, the last paragraph of Section 2.1 and Step 7 in Table 2-1 (DQOs) have been revised to reference FSP/QAPP Table 4-5, the project performance measurement criteria, and FSP/QAPP Tables 4-6 and 4-7, the table presenting comparisons of laboratory detection limits to the human health screening levels for soil and groundwater, respectively.*

A#2 - For analytes where project criteria are lower than lab detection limits, a discussion on how these results will be used in the final evaluation should be added to the SAP.

P4 Response: *The following paragraph has been added to the end of the text in Section 5.3 of the FSP/QAPP, "Table 4-5 notes that there are the method detection limits for several target compounds are greater than the human health screening levels for soil and groundwater. The specific compounds are identified with footnote "c" on Table 4-6 for soil (four SVOCs) and on Table 4-7 for groundwater (seven VOCs and 11 SVOCs). The uncertainty related to this will be addressed as part of the human health risk assessment."*

A#3 - The level of effort and information provided for the organic specs should be the same as for other constituents addressed in the QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. If P4 wishes further clarification on this issue, the A/T believes that this would be best handled in a conference call that includes A/T and P4 data quality expert staff. Furthermore, at P4's discretion, these specs may be submitted as an addendum to the QAPP/QAPP Addendum rather than just for the Ballard Shop project so they can be used for other future work, as needed. If P4 wishes to amend the QAPP/QAPP Addendum with these specs or any other information, the exact means and process to do so should first be agreed to by the A/T in order to ensure appropriate version control.

P4 Response: *Section 3.4.4 has been added to provide specific detail for the laboratory hard-copy data deliverables for VOCs, SVOCs, and PCBs. The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the organic methods as has been provided in the QAPP Addendum for inorganic constituents. The data validation report templates (now provided in Appendix C of the FSP/QAPP) have been revised to reference the FSP/QAPP tables. Please note that the target compounds list is too extensive to concisely summarize the control limits in the text of the templates. P4 does not anticipate future investigations for organic constituents and prefers to provide the sampling and analytical criteria for these organic methods in this stand-alone SAP.*

A/T Follow-up Comment: The validation templates in the Addendum contained the final report to be used by LDC (or any other validator). Please confirm that report is being provided for the Ballard Shop study.

P4 Follow-up Response: P4 confirms that the final data validation reports, which will be based on the templates provided in Appendix C of the FSP/QAPP, will be provided to the A/T in the RI/FS report.

Specific Comments, Appendix A

A#4 - Section 2.0, page 2-1, 1st paragraph. The same comment regarding editorial comment as made above for Section 1.1, page 1-3, 1st complete paragraph of the SAP applies here in the FSP, as well.

P4 Response: *The first paragraph in section 2 of the FSP now reads:*

"This section provides brief background information related to the Ballard Mine Shop investigation. Additional program background and objective details are provided in Section 1.1 of the SAP and the RI/FS Work Plan. Because this facility was operated as a maintenance shop for heavy trucks and mining equipment from approximately 1952 to 1989 for both the Ballard and Henry Mines there may have been incidental spills of oil, polychlorinated biphenyls (PCBs), solvents, and other hydrocarbons (i.e., lubricants, fuel, etc.). As a result, P4 has agreed to collect additional soil and groundwater samples to confirm the current conceptual model for the shop area. Although, because the hydrocarbons that may have been released in the shop and on surrounding surface soils are biodegradable, today there likely would be only residual organic concentrations and degradation products remaining."

A#5 - Section 3.1.1., page 3-2, 1st full paragraph. For PCBs, sample depths are not specified. The text should specify sample depths and describe contingency sampling at depth if field data indicate that a release extends significantly beyond the first few feet of surface soils.

P4 Response: *Section 3.2 "Sample Collection Procedure" discusses where and how the soil samples will be collected during the Ballard Mine Shop investigation. The final paragraph in Section 3.2.2 indicates that two soil samples will be collected at each PCBs boring location; the first below the slag/native soil interface and the second at 4-5 feet below the existing ground surface. Additional information has been added to Section 3.2.2 (Soil Investigation (PCBs)) to indicate the boreholes will be extended if visual contamination or odors occur at the second sample depth. This section now reads:*

"Soil Investigation (PCBs). *Shallow soil samples also will be collected in the two identified transformer locations and analyzed for PCBs. Soil borings SB-5 and SB-6 will be located next to the identified transformer areas to the west and south of the shop building as depicted on Figure 3-1. The HSA drill rig will be utilized to advance these two soil borings within the alluvial material to the required depth (refer to SOP-1). Soil samples will be collected with a CME (or similar) split barrel sampling system or split-spoon samplers. Samples will be collected at the native soil interface, which is assumed to be approximately six to 12 inches bgs. A second sample interval will be collected at a depth of four to five feet bgs (approximately three to four feet below native soil).*

Should visual contamination or odors be detected in the second sample interval, then the boreholes will be continuously cored until no contamination indicators are observed or groundwater is reached. A third and final soil sample then will be collected just beneath the identified contamination or just above the water table to confirm the vertical extent of contamination.

The soil samples will be collected with a clean stainless steel spoon or scoop and placed in an appropriately-sized container as provided by the laboratory. Sampled soil intervals will be logged in general accordance with USCS protocol. The soil samples will be analyzed for PCBs according to the methods described on Table 3-2 and in Section 4.3."

A#6 - Section 3.2.2., page 3-5, 2nd full paragraph. Similar to a previous comment on field screening soil samples, the text should describe how the split spoon samples will be screened, e.g., using headspace or some other means.

P4 Response: *Section 3.2 has been revised to indicate how each soil core interval from the split spoon sampler will be screened in the field. Please refer to the revisions in Section 3.2.2.*

A#7 - Section 3.2.2, page 3-7. The temporary well installation description indicates that wells will not be developed. Explain the rationale for not developing the wells or provide a description of the development methods.

P4 Response: *The temporary wells will not be developed because we are planning to abandon them once the groundwater sampling and water level collection activities are completed. As described, we are planning to collect grab groundwater samples from each of these boreholes that may be turbid. As a result, these samples would indicate "worse case" contaminant conditions if contaminants are present because the sediments in the groundwater, in addition to the groundwater, could contain the constituents of potential concern. However, these temporary wells will be developed if contamination is detected above relevant benchmarks and they are needed for further monitoring at the site. Well development procedures will be described in a subsequent addendum to this SAP if it is necessary.*

A#8 - Sections 4.3 and 4.4, tables 4.8, 4.9, 4.10. The analytical and data validation specs introduced here are not consistent with the specs for other constituents in the parent QAPP and QAPP Addendum. P4 should revise the method specific analytical specs for lab analyses and data validation for the organic constituents to be consistent with and equivalent to those prepared for other constituents addressed in the QAPP Addendum. As noted above under general comments, if new method specific specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: *Please refer to the response to Comment A#3 above regarding the addition of Section 3.4.4 to address specific requirement for hard-copy laboratory deliverables for VOCs, SVOCs, and PCBs and existing analytical specificity provided in Tables 4-8 through 4-13. Section 4.5 has been revised to provide additional "Reason Codes" for organic methods (and these reason codes have been added to the data validation report templates). The existing Tables 4-8 through 4-13 provide the equivalent level of detail for the validation of organic data as has been provided in the QAPP Addendum for inorganic data. The data validation templates have been revised to include additional level of detail.*

A/T Follow-up Comment: To clarify, the data validation requirements/specs and data validation report templates need to be identical to the Addendum with method specific elements.

P4 Follow-up Response: The revised templates include method specific elements and are included in the draft final SAP.

A#9 - Section 4.5 and Appendix B. The validation level descriptions and the proposed level of effort are not consistent with the specs and level of effort agreed to in the parent addendum. Consistent with the previous validation efforts since the development of the QAPP Addendum, all data need to be 'validated' and flagged by reviewing all QC summary data for all QC parameters (to include initial calibration, continuing calibration, tuning, internal standards, interference checks, serial dilutions, etc.). Additionally 10% of the data needs to be reviewed for raw data. EPA data validation functional guidance should be used for reviewing and flagging the data. As noted above under general comments, if new method specific data validation specs are developed, they may be added to the parent QAPP addendum upon A/T approval.

P4 Response: *With revisions to the Reason Codes (see response to Comment A#8), revised Section 4.5 provides the equivalent level of detail for the validation of organic data as has been provided in the QAPP Addendum for inorganic data. The data validation templates (now provided in Appendix C) have been revised to provide additional detail.*

A/T Follow-up Comment: Please see the A/T response to comment A#8, above.

P4 Follow-up Response: *The revised templates include method specific elements and are included in the draft final SAP.*

Editorial Comments on Appendix A

A#10 - Section 3.1.2, page 3-3, paragraph 1, line 3. Delete *of*.

P4 Response: *Agreed.*

A#11 - Section 3.2.2, page 3-6, paragraph 4 (last), line 3. Delete *than*.

P4 Response: *Agreed.*

A#12 - Section 3.2.2, page 3-7, paragraph 3, line 1. Delete *of*.

P4 Response: *Agreed.*

A#13 - Section 3.2.3, page 3-8, paragraph 4, line 2. Delete *in*.

P4 Response: *Agreed.*

A#14 - Section 3.2.5, page 3-10, paragraph 2, line 2. Delete first *type of*.

P4 Response: *Agreed.*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
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April 18, 2011

Barry Koch
Special Projects Lead – Mining
Monsanto Company
P.O. Box 816
Soda Springs, Idaho 83276

Re: Comments on P4's *Ballard Mine Shop Investigation Sampling and Analysis Plan Revision 1 Draft*, prepared for P4 Production by MWH, April 2011.

Dear Mr. Koch,

The Agencies and Tribes (A/T) have reviewed the above referenced deliverable submitted by P4. This work product was developed pursuant to the 2009 RI/FS Settlement Agreement. Our comments are enclosed.

We will be available to discuss this matter, either by conference call, or as necessary during subject-specific meetings. If no further discussion is needed, it's our understanding that you will submit an electronic version of the revised deliverable for our final review and approval. I can be reached at 208-378-5763 or electronically at tomten.dave@epa.gov.

Sincerely,

//s//

Dave Tomten
Remedial Project Manager

Enclosure

cc: Cary Faulk, MWH (electronic version only)
Vance Drain, MWH (electronic version only)
Mike Rowe, IDEQ
Mary Kaufman, FS
Jim Alexander, USDA
Forest Service - Enoch Valley Site Record



Jeff Cundick, BLM
Sandi Fisher, US FWS
Kelly Wright, Shoshone Bannock Tribes
Susan Hanson (for the tribes)
Colleen O'Hara, BLM (electronic version only)
Eldine Stevens, BIA (electronic version only)
Tim Mosko, CH2MHill (electronic version only)
Sherri Clark, FS (electronic version only)
Charles Allbritton, EPA Records Center (electronic version only)

Comments on P4's *Ballard Mine Shop Investigation Sampling and Analysis Plan* Revision 1 Draft, prepared for P4 Production by MWH, April 2011.

April 18, 2011

General Comments

In the A/T's March 15, 2011 general comment on Revision 0 of the Ballard Shop SAP, the A/T noted that the organization and format of the Ballard Shop SAP was not consistent with that recommended in EPA's G-5 guidance. Although no further response to this comment is needed at this time, we want to reiterate that it is the A/T's preference to have all QAPP elements contained in a single document. This approach allows for the most expeditious review.

Specific Comments

Appendix A, Section 3.4.4. The extensive text here falls short of capturing all method specifics. All laboratory packages should be at the full level equivalent to CLP packages irrespective of the level of validation. This section can be replaced with a statement to the effect that all laboratory data packages will be equivalent to CLP packages providing the same information as the CLP packages in a manner that is comprehensible to chemists outside the lab. EPA references should also be added.

Appendix A, Section 4.5. The text needs to be preceded by a statement that the data will be validated at the following two levels of effort per templates provided in appendix:

- 10 % of the data will be validated fully per EPA functional guidance (provide method specific EPA functional guidance references here) to include raw data review.
- 90 % of the data will be reviewed per data QC summaries only (no raw data reviews) to cover all QC parameters identified in the EPA functional guidance (e.g. initial calibration, initial calibration verification , continuing calibration, tuning, internal standard as applicable to different methods).

Appendix A, Table 4.10. For method 8082, add QC parameters relating to compound ID verification.

Appendix C of Appendix A (FSP/QAPP Data Validation Templates).

For method 8082, add QC parameters relating to compound ID verification.

April 20, 2011

A/T's Comments on P4's *Ballard Mine Shop Investigation Sampling and Analysis Plan Revision 1 Draft*, prepared for P4 Production by MWH, April 2011.

General Comments (GC)

GC#1 - In the A/T's March 15, 2011 general comment on Revision 0 of the Ballard Shop SAP, the A/T noted that the organization and format of the Ballard Shop SAP was not consistent with that recommended in EPA's G-5 guidance. Although no further response to this comment is needed at this time, we want to reiterate that it is the A/T's preference to have all QAPP elements contained in a single document. This approach allows for the most expeditious review.

P4's Response: *Understood.*

A/T's Specific Comments (SC):

SC#1 - Appendix A, Section 3.4.4. The extensive text here falls short of capturing all method specifics. All laboratory packages should be at the full level equivalent to CLP packages irrespective of the level of validation. This section can be replaced with a statement to the effect that all laboratory data packages will be equivalent to CLP packages providing the same information as the CLP packages in a manner that is comprehensible to chemists outside the lab. EPA references should also be added.

P4's Response: *The level of detail provided in Section 3.4.4 for the contents of the laboratory data package is consistent with that provided in the Supplemental Mine Waste Rock Dump and Facility Soil and Vegetation Characterization SAP. This is the SAP the A/Ts requested P4 use as a template for future QAPPs (please note that the method-specific information is summarized on Tables 4-8 through 4-10). Section 3.4.4 indicates that all data reports (regardless of the level of validation) will be submitted with Stage 4 deliverables (that is, full raw data reports with all required summary forms). EPA reference for the CLP SOW for Organic Analysis (USEPA, 2005) has been added to the current document.*

SC#2 - Appendix A, Section 4.5. The text needs to be preceded by a statement that the data will be validated at the following two levels of effort per templates provided in appendix:

- 10 % of the data will be validated fully per EPA functional guidance (provide method specific EPA functional guidance references here) to include raw data review.
- 90 % of the data will be reviewed per data QC summaries only (no raw data reviews) to cover all QC parameters identified in the EPA functional guidance (e.g. initial calibration, initial calibration verification , continuing calibration, tuning, internal standard as applicable to different methods).

P4's Response: *The text has been added as requested.*

SC#3 - Appendix A, Table 4.10. For method 8082, add QC parameters relating to compound ID verification.

P4's Response: *Table 4-10 has been revised to include criteria for compound identification.*

SC#4 - Appendix C of Appendix A (FSP/QAPP Data Validation Templates).
For method 8082, add QC parameters relating to compound ID verification.

P4's Response: *The Method 8082 data validation report template has been revised to include a section and criteria for compound identification.*



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
IDAHO OPERATIONS OFFICE
1435 N. Orchard St.
Boise, Idaho 83706

April 28, 2011

Barry Koch
Special Projects Lead – Mining
Monsanto Company
P.O. Box 816
Soda Springs, Idaho 83276

Re: Conditional Approval of *Ballard Mine Shop Investigation Sampling and Analysis Plan, Revision 2 Draft*, prepared for P4 Production by MWH, April 2011.

Dear Mr. Koch,

The Agencies and Tribes (A/T) have reviewed the above referenced deliverable submitted by P4. This work product was developed pursuant to the 2009 RI/FS Settlement Agreement. All previous A/T comments have been addressed. The following comments are new and should be easy to incorporate into the final SAP. Because these comments appear to be minor, and additional comments are not anticipated, we are now providing conditional approval of the SAP.

If no further discussion is needed, it's our understanding that you will submit a final version of the deliverable to the A/T for our records and final approval. Please contact me if you have questions or concerns. I can be reached at 208-378-5763 or electronically at tomten.dave@epa.gov.

Sincerely,

//s//

Dave Tomten
Remedial Project Manager

Enclosure

cc: Cary Faulk, MWH (electronic version only)
Vance Drain, MWH (electronic version only)
Mike Rowe, IDEQ
Mary Kaufman, FS



Jim Alexander, USDA
Forest Service - Enoch Valley Site Record
Jeff Cundick, BLM
Sandi Fisher, US FWS
Kelly Wright, Shoshone Bannock Tribes
Susan Hanson (for the tribes)
Colleen O'Hara, BLM (electronic version only)
Eldine Stevens, BIA (electronic version only)
Tim Mosko, CH2MHill (electronic version only)
Sherri Clark, FS (electronic version only)
Charles Allbritton, EPA Records Center (electronic version only)

Comments on P4's *Ballard Mine Shop Investigation Sampling and Analysis Plan, Revision 2 Draft*, prepared for P4 Production by MWH, April 2011.
April 28, 2011

Specific Comments

Appendix A, Table 4.10. In the "Compound Identification" row, "Corrective Action/Lab Flagging Criteria" column, add text to the effect that qualification of detects will be carried out as described under validation flagging. The laboratory should not be reporting detects as ND when these criteria are not met. The laboratory should use professional judgment in qualifying detects as is described under the validation column.

Editorial Comments

Appendix A, Section 4.5, page 4-13, bullet 2, line 3. Delete space between verification and the subsequent comma.

Appendix A, Section 4.5, page 4-13, bullet 2, line 4. Delete "and."

Appendix C of Appendix A, Laboratory Data Consultants, Inc., Data Validation Report, Polychlorinated Biphenyls by GC SW-846 Method 8082, page 5. There are two sections labeled IX.

Response to Comments on P4's *Ballard Mine Shop Investigation Sampling and Analysis Plan* included A/T Conditional Approval Letter, *Revision 2 Final*, prepared for P4 Production by MWH, April 2011.

April 28, 2011

Specific Comments

Appendix A, Table 4.10. In the "Compound Identification" row, "Corrective Action/Lab Flagging Criteria" column, add text to the effect that qualification of detects will be carried out as described under validation flagging. The laboratory should not be reporting detects as ND when these criteria are not met. The laboratory should use professional judgment in qualifying detects as is described under the validation column.

P4's Response: *Text in reference cell was changed from, "Report target as not detected (ND) if criterion not met" to "Report target if %D criterion not met, but report QC failure with the result and note in the case narrative."*

Editorial Comments

Appendix A, Section 4.5, page 4-13, bullet 2, line 3. Delete space between verification and the subsequent comma.

P4's Response: *The space has been deleted.*

Appendix A, Section 4.5, page 4-13, bullet 2, line 4. Delete "and."

P4's Response: *The typo has been corrected.*

Appendix C of Appendix A, Laboratory Data Consultants, Inc., Data Validation Report, Polychlorinated Biphenyls by GC SW-846 Method 8082, page 5. There are two sections labeled IX.

P4's Response: *The second Section IX has been changed to Section X, and the last section has been changed to Section XI.*

ATTACHMENT 2
A/T APPROVAL OF 2014 ADDITIONAL BALLARD SHOP WELL SAMPLES



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 10
IDAHO OPERATIONS OFFICE
950 West Bannock, Suite 900
Boise, Idaho 83702

April 17, 2014

Rachel Roskelley
Sr. Environmental Engineer
Monsanto Company
Soda Springs Operations
1853 Highway 34
Soda Springs, Idaho 83276

Re: Approval of Proposed Ballard Shop Monitoring Well Sample Collection in Addition to the P4 Long-Term Surface Water and Groundwater Monitoring Plan – Draft Rev 0 – 2014, dated April 7, 2014.

Dear Ms. Roskelley,

The Agencies and Tribes (A/T) have reviewed and approve the above referenced memorandum describing the proposed re-sampling of two monitoring wells for a limited list of analytes at the Ballard Shop area. The sampling would be conducted according to the methods and procedures described in a previously approved sampling and analysis plan. This deliverable is submitted pursuant to the Administrative Settlement Agreement and Order on Consent/Consent Order for Performance of Remedial Investigation and Feasibility Study at the Enoch, Henry, and Ballard Mine Sites in Southeastern Idaho (or 2009 AOC).

Please produce and distribute a final version of the plan. As we have discussed in the past, some agencies have provided direction that it is acceptable to provide an electronic version of the final deliverable on a CD (rather than both a hardcopy and electronic copy). Please contact me if you have questions. I can be reached at 208-378-5763 or electronically at tomten.dave@epa.gov.

Sincerely,

//s//

Dave Tomten
Remedial Project Manager

Attachment

cc: Cary Faulk, MWH (electronic version only)
Vance Drain, MWH (electronic version only)
Mike Rowe, IDEQ – Pocatello
Sandi Fisher, US FWS - Chubbuck

Kelly Wright, Shoshone Bannock Tribes
Susan Hanson (for the tribes)
Talia Martin, Shoshone Bannock Tribes (electronic version only)
Mary Kaufman, FS – Pocatello (electronic version only)
Colleen O'Hara, BLM (electronic version only)
Eldine Stevens, BIA (electronic version only)
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Charles Allbritton, EPA Records Center (electronic version only)